

# METALLURGIA

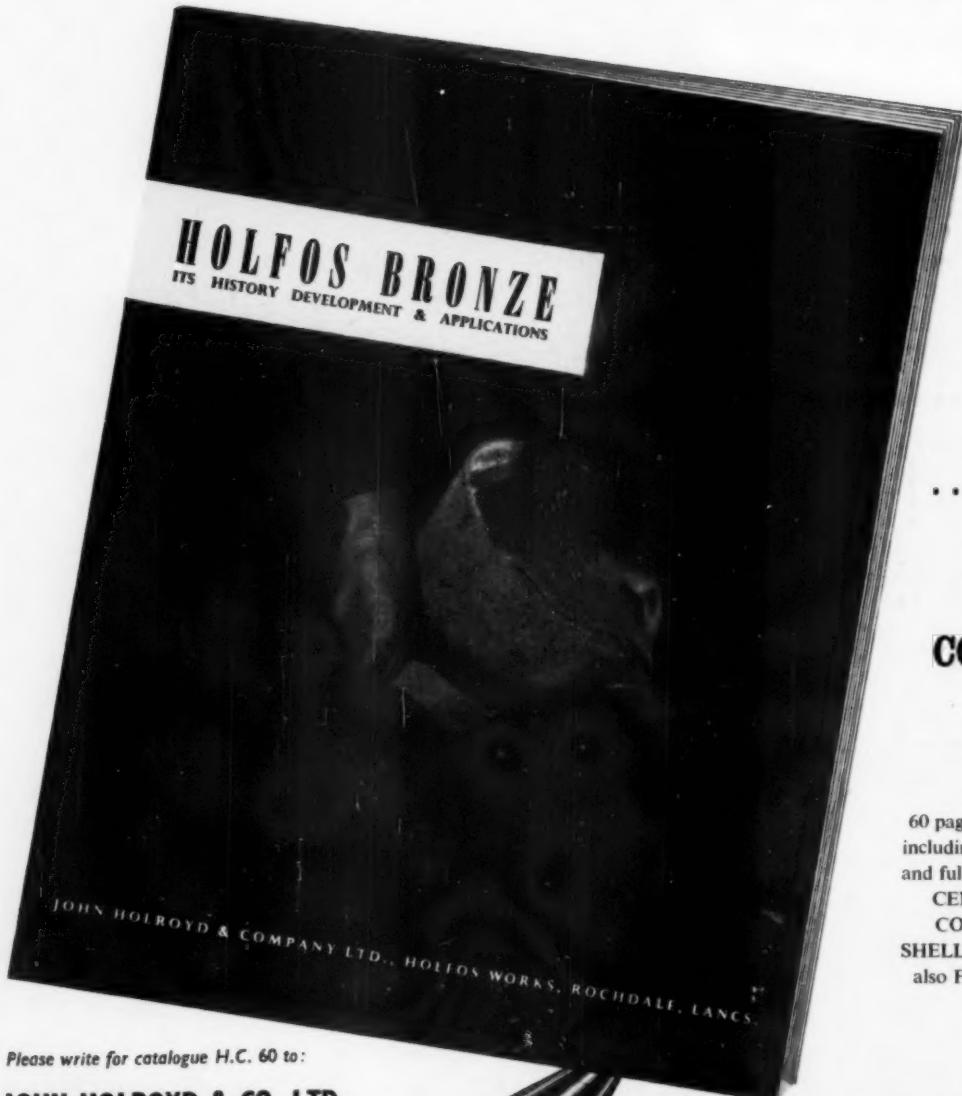
THE BRITISH JOURNAL OF METALS

Vol. 62 No. 374

DECEMBER, 1960

Monthly: Two Shillings and Sixpence

## A new publication . . .



... OF VITAL  
INTEREST  
TO ALL  
CONCERNED  
WITH  
BRONZE

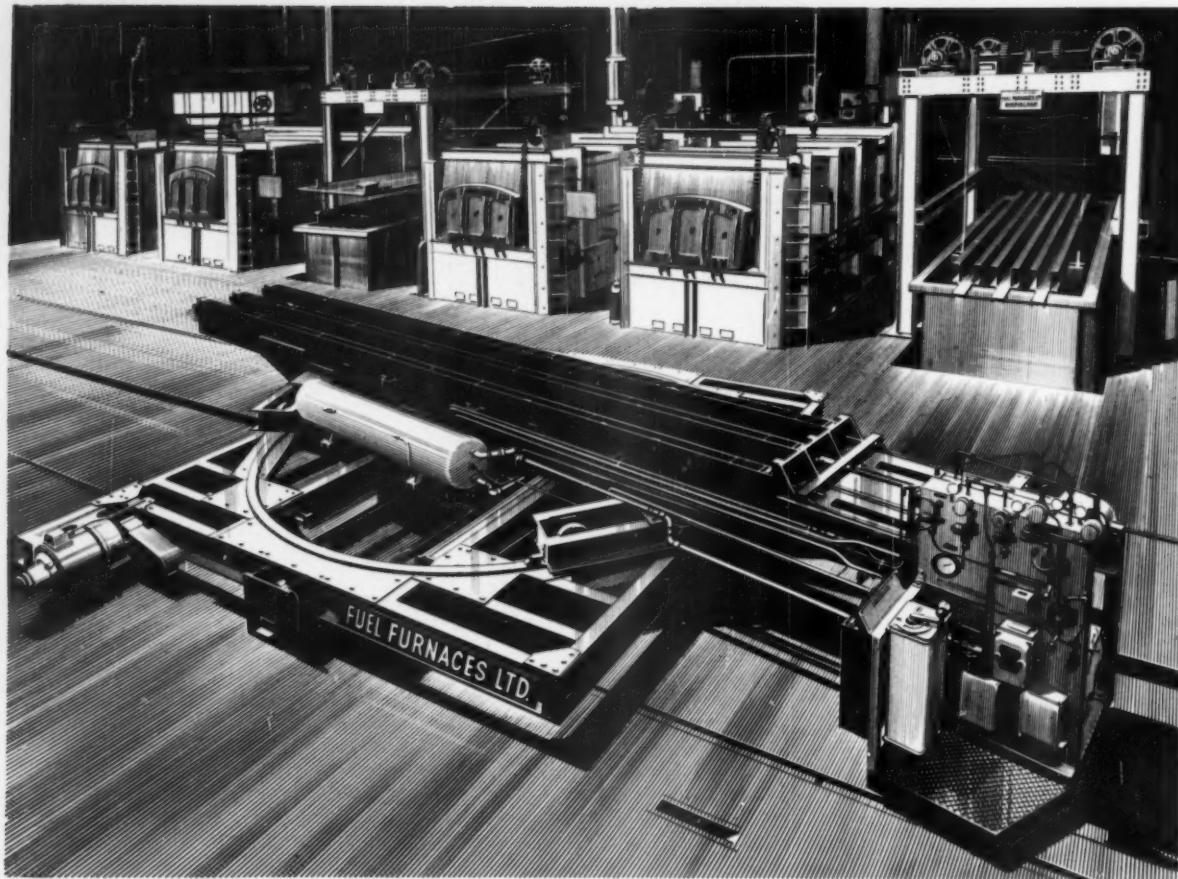
60 pages of informative reading,  
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SHELLCAST Bronze Materials ;  
also FINISHED MACHINED  
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Impression by courtesy of The Kirkstall Forge Engineering Ltd.

heat  
treatment  
of  
steel bars and coils

high velocity recirculating atmosphere

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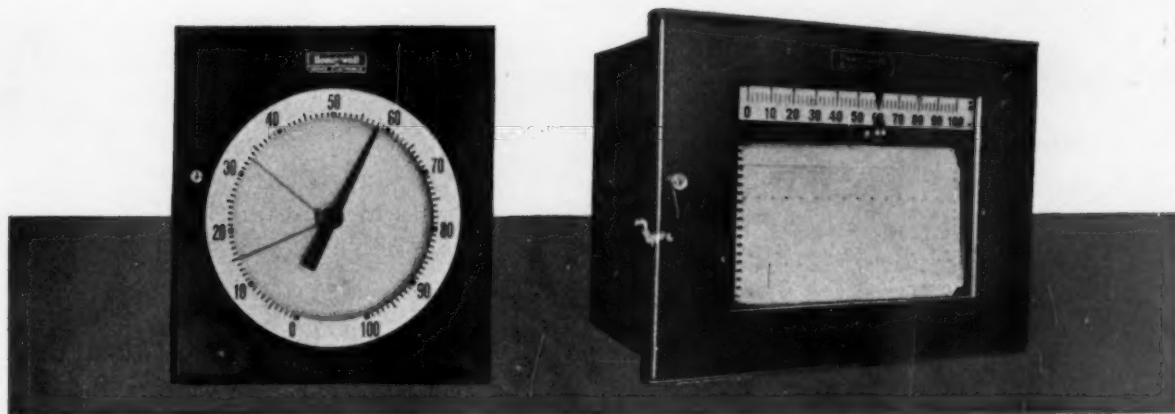
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# New modular design...

*makes Electronik potentiometers  
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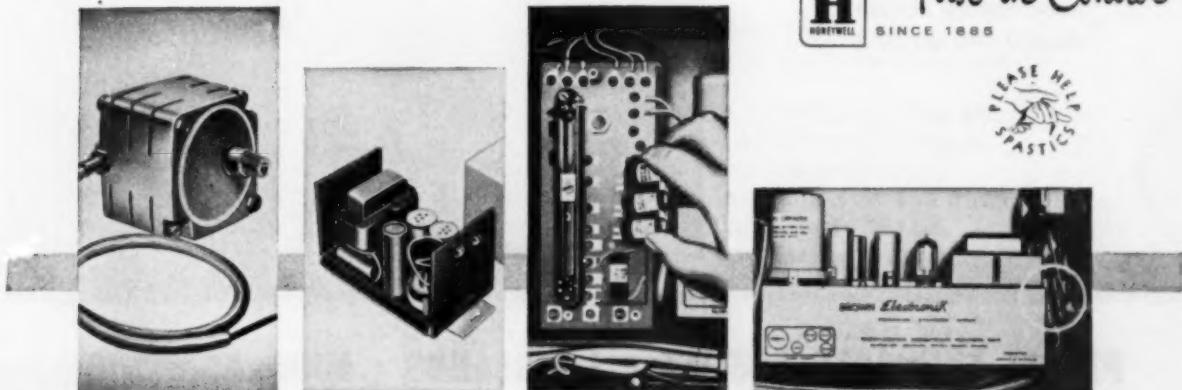
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WAXlow 2333.

## Honeywell



First in Control

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1 Balancing Motor

2 Constant Voltage Module

3 Measuring Circuit Module

4 Amplifier Module

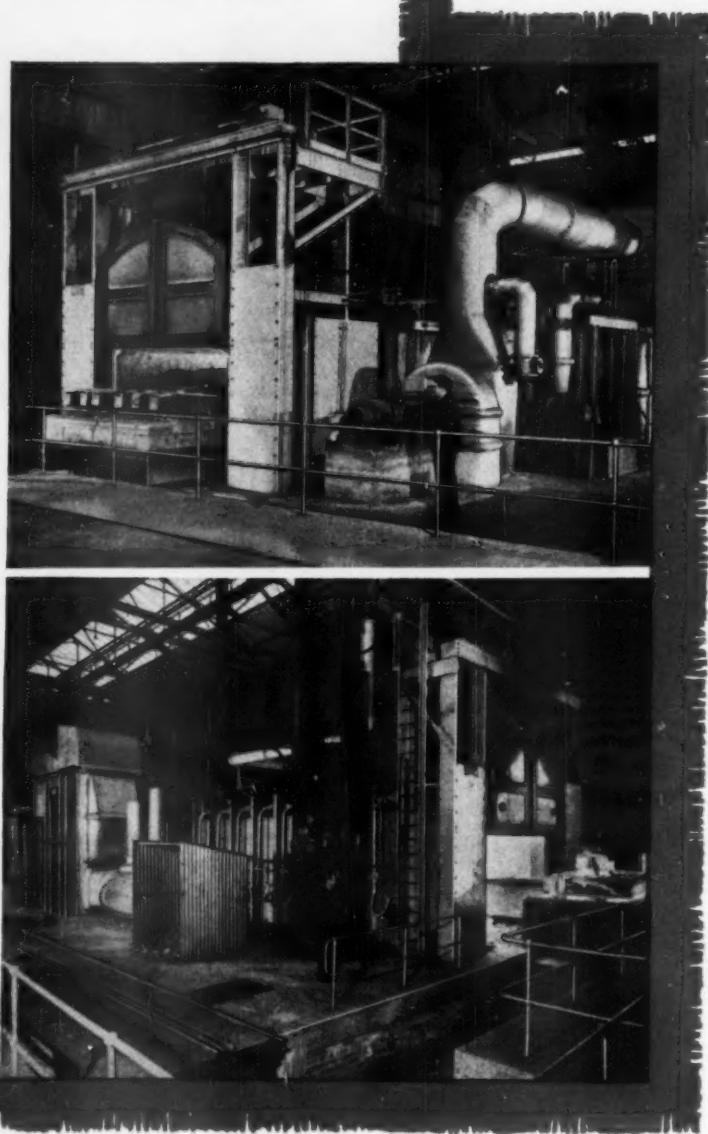
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Illustrated is a Gas-fired Furnace supplied to Jessop Saville Ltd. Brightside Works, Sheffield, for reheating alloy steel ingots prior to rolling.

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Open Hearth Furnaces  
Soaking Pits of all types  
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Furnaces for Aluminium Melting, Coil Annealing and Slab Re-heating  
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F 155



# WE'RE IN THE PICTURE

You can't see us—not at this distance, but we are there just the same. In all manner of ways we make our contribution to human flight.

Here are some examples. T1 and T2 thermocouple alloys play their part in engine temperature and performance. Nichrome\* heating elements do their job in the food containers. In radar and radio equipment you will find our glass-sealing alloys, cathode and high-purity nickel, as well as superfine resistance wires.

Over the years manufacturers of apparatus for aircraft have come to put their faith in British Driver-Harris high nickel alloys. We will be glad to let you have technical information.

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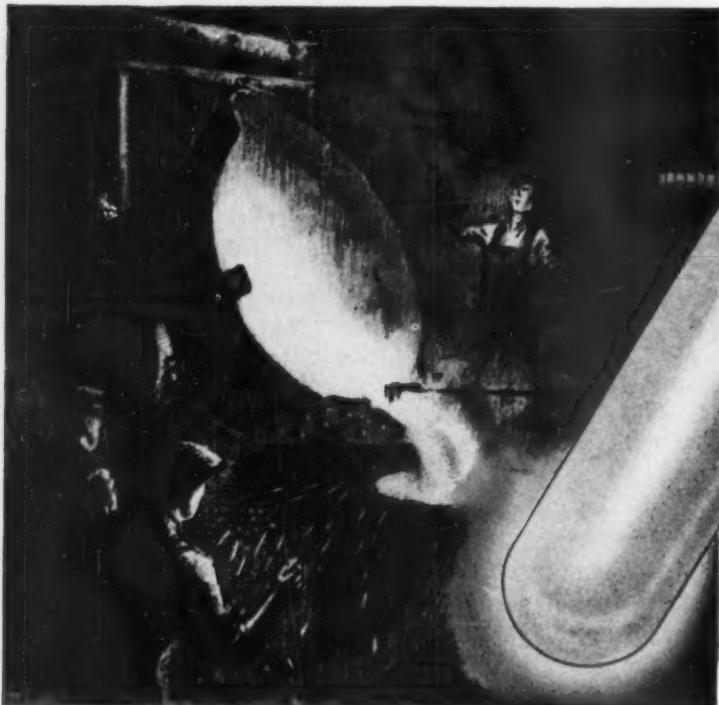
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A BRITISH PRODUCT  
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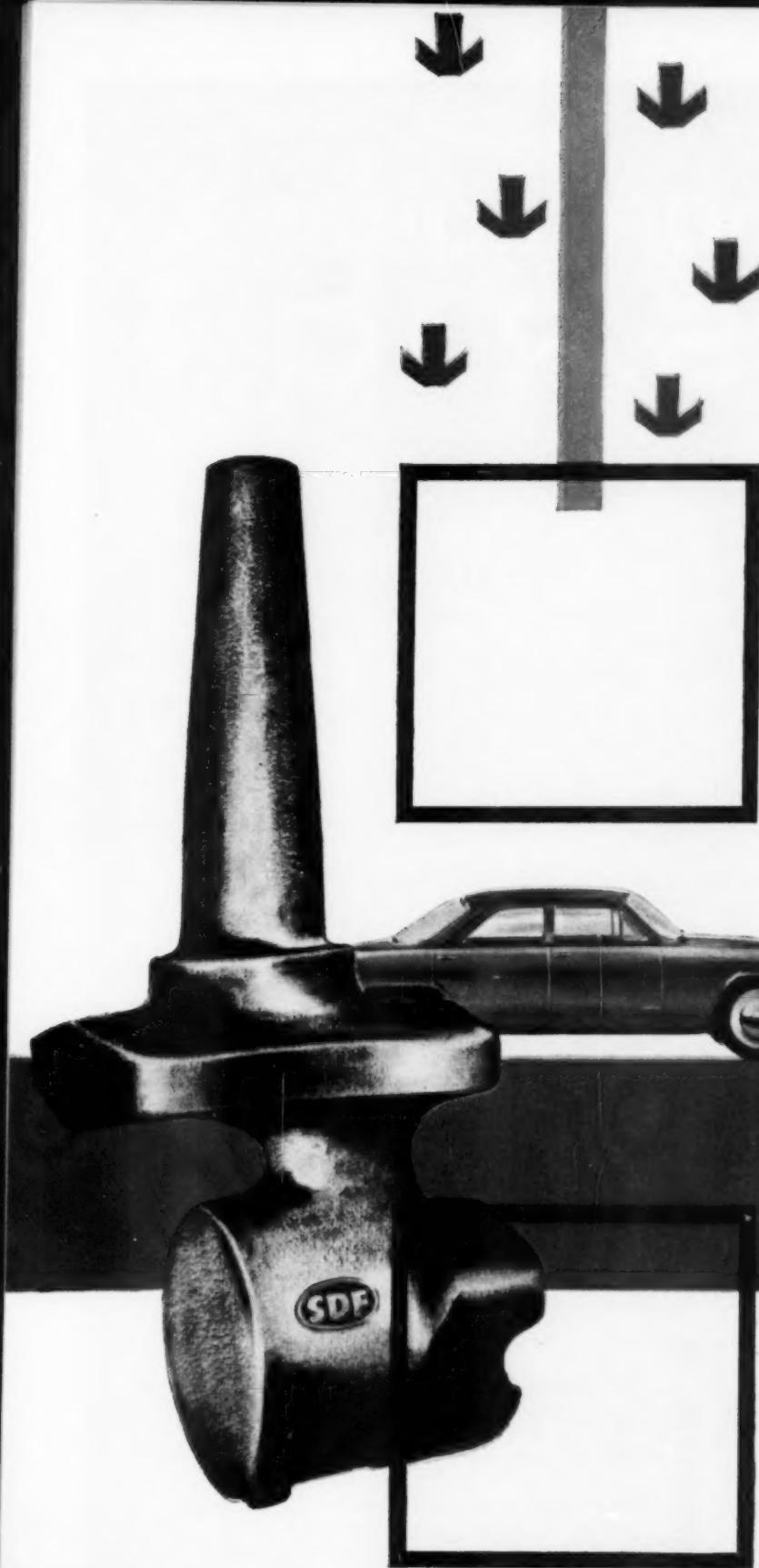
METALLURGIA, December, 1960



## VITREOSIL DIPPING TUBES

Casting temperatures are *critical*! Vitreosil, pure fused silica, sheaths used in the Quick Immersion Thermocouple Technique have obviated erratic judgement of casting temperatures in modern steelworks and foundries all over the world. A single dip into the molten metal gives the true temperature in seconds.

No more reject castings through too hot, or too cold, tapping, if you turn to Vitreosil — for accurate, up-to-date control.



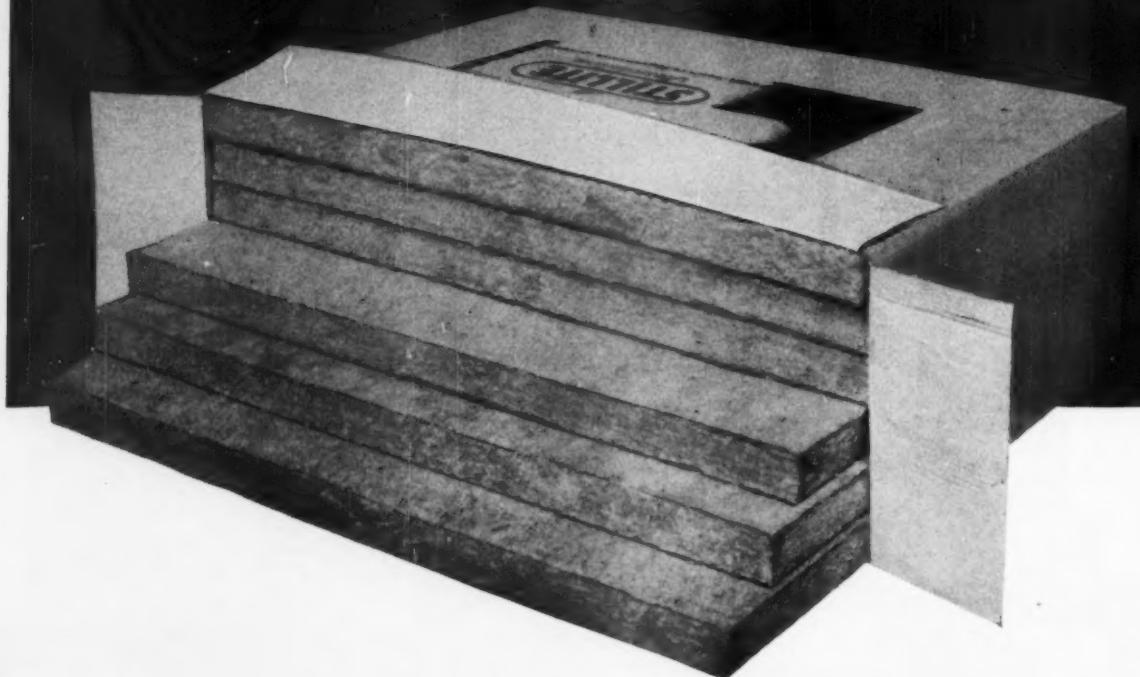
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**The stamp of quality**



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**CUT YOUR LOSSES  
with S.R.10**



The furnace industry is well acquainted with THERBLOC\* for insulation up to 1,500°F. Where lower temperatures are involved however, for example core stoves and paint booths, consider S.R.10 Mineral Wool slabs. An inexpensive, yet, valuable material not only for maintaining temperatures and reducing fuel bills but also to make working conditions more acceptable to operators.

\* THERBLOC is a highly efficient Mineral Wool Slab which is being used extensively in place of diatomite as a backing insulant at interface temperatures up to 815°C. Write for Technical Data No: 5 "THERBLOC for furnace and stove insulation."

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To increase our output of Industrial Furnaces we have acquired a modern Factory in Bletchley, Buckinghamshire, to which we have transferred the Furnace building side of our business.

The factory covers an area of some 15,000 sq. ft. and we can therefore now handle much larger Furnaces than before. The Works has been equipped with up-to-date Machine Tools, and we have excellent loading and unloading facilities which include an overhead runway with an electric lifting block of 5-ton capacity.

We carry a wide range of refractories which enables us to handle urgent repair work, and give prompt delivery for new equipment. We shall be pleased to receive inquiries for Gas, Oil and Electric Furnaces, Salt Baths, Air Circulators, etc.

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# STEIN Refractories

THE REFRACTORY BRICK FOR SOAKING PIT  
Hearth, lower side walls and Ingot Head Level

## STEIN 73

For exceptional durable service  
Stein 73 is unrivalled in its resistance to damage  
by abrasion and from slag attack.

*You are invited to consult our long experience on all refractory problems*

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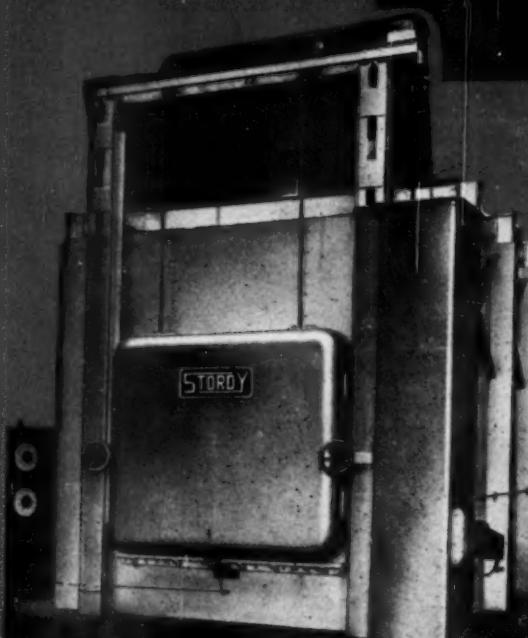
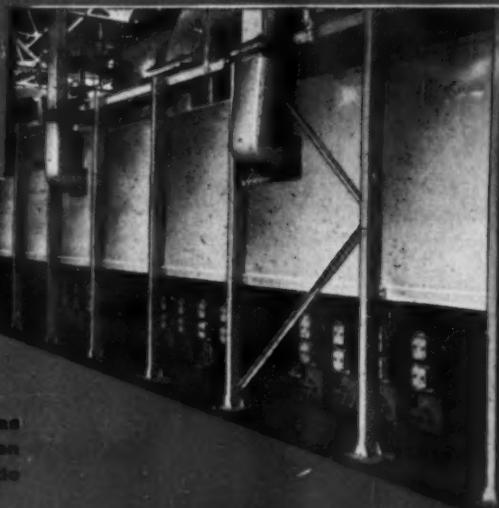
**STORDY****HEAT TREATMENT FURNACES**

Illustration above: Electrically heated, air circulated batch-type furnace for aluminum. Working temperature 800°C, the furnace being complete with electric heater batteries and circulating fans, designed for easy removal.

Illustration right: High temperature heat treatment oven for television tubes. The oven has a heated length of 206' 0", being provided with eleven combustion chambers, circulating fans and thermocouple controls for a maximum temperature of 480°C.

The range of Stordy heat treatment furnaces include batch, pit and continuous types, heated by oil, gas or electricity, with indirect firing when required. The Stordy system of forced air circulation is unique in its efficiency and in providing rapid heating up rates and uniform heating conditions throughout the chamber with maximum fuel economy.

Stordy heat treatment plant includes all the necessary materials handling equipment—robust charging and discharging machine, conveyors, transfer tables, loading and unloading racks.

**DESIGNED AND FABRICATED BY****STORDY**

Stordy have full manufacturing facilities at their new Works and design staff, technical and service engineers in the field combine to provide a completely integrated furnace design and building service.

**STORDY ENGINEERING LIMITED**

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Temperature range 600-1550 C  
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Photograph by G. H. H. of the Reversing Hot and Cold Break-down Mill or Non-Ferrous Products

**THE BRIGHTSIDE FOUNDRY & ENGINEERING CO. LTD.**

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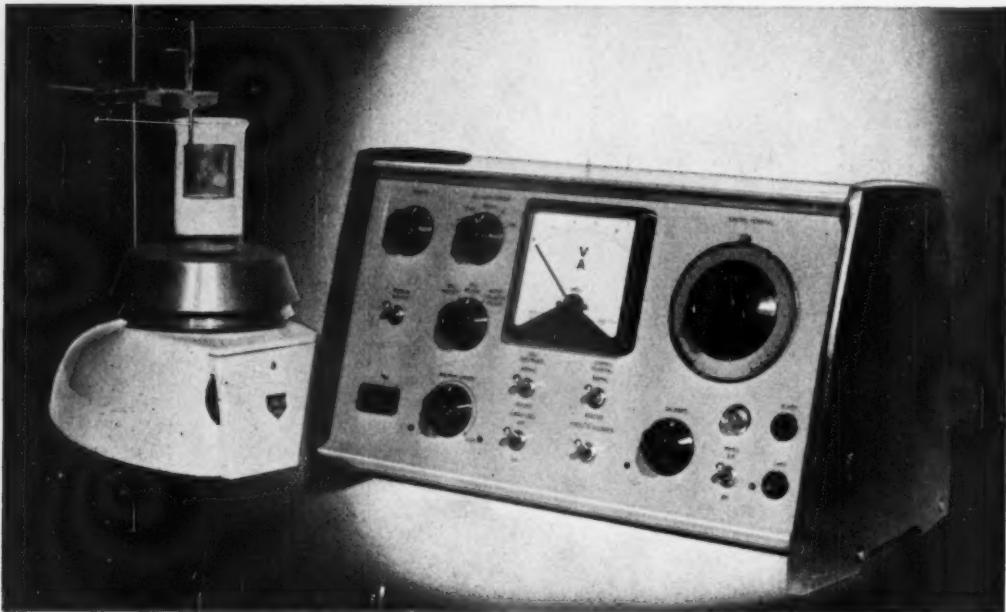
**A 1654**

## **Wadsworth Controlled Potential Electro-Depositor**

ELECTRO-DEPOSITION has long been recognised as an analytical and separative technique for solutions containing metallic ions. The method may be applied where the ionic concentrations of the metals present in solution vary widely, separation being best achieved where the potential of the working electrode relative to the solution is controlled, using a reference electrode as described

passing through the cell in such a sense that the potential error tends towards zero.

In order to ensure that thermal drift in the control amplifier is held at a very low level, a water-cooled heat sink is employed to maintain the transistors at a substantially constant temperature. The accuracy of control attained may be judged from the fact that the incremental error in electrode



independently by Sand & Fischer. Manual control of the electrode potential, although possible, is tedious and time consuming, with the result that automatic control circuits have been developed by a number of workers.

The Southern Analytical controlled potential electro-depositor is based upon a design due to N. J. Wadsworth of R.A.E., Farnborough, and is intended primarily for use with platinum electrodes. The circuit is fully transistorised in the interests of compactness and reliability and is capable of supplying a maximum controlled current of 10 amperes to the electrolysis cell. In operation, the required cathode potential is preset upon a built-in high grade potentiometer. Any error between the preset potential and that of a reference half-cell (e.g. saturated calomel), mounted immediately adjacent to the cathode, is applied to an amplifier which controls the current

potential as a function of cell current has a mean value of 1.6 millivolts/ampere; thus, when the electrolysis current has fallen to the low levels obtaining towards the end of a deposition, extremely close control is established. It is noteworthy that the potential defined by the built-in potentiometer cannot be exceeded as cell current cuts off at zero potential error.

The apparatus is supplied complete with leads for connection to the user's own electrode assembly. If desired however, suitable standard accessory equipment can be provided.



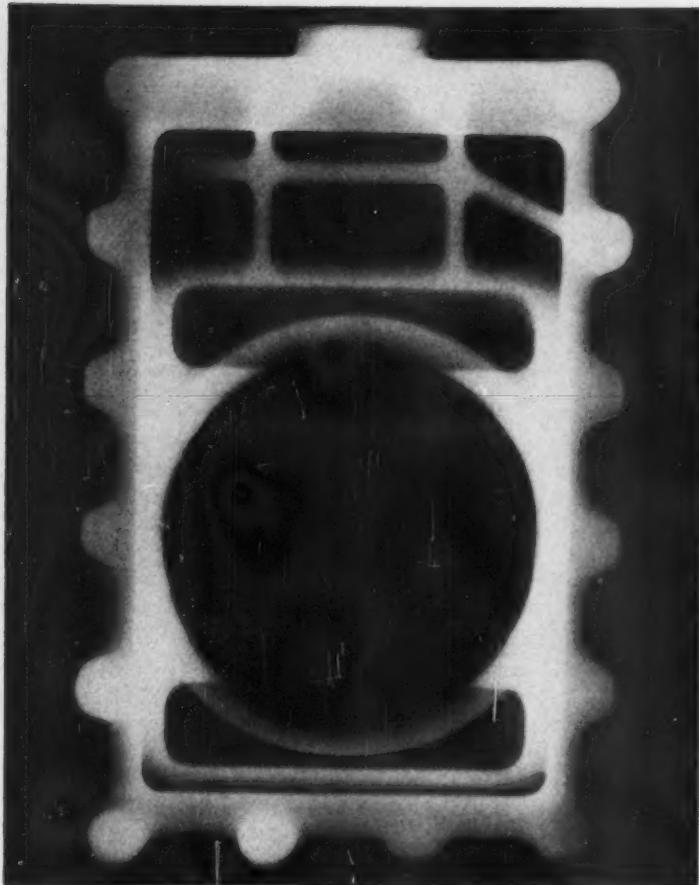
# LARGE G.E.C. FURNACES

G.E.C. Electric Furnaces of the continuous type are in operation with individual outputs exceeding 10 tons per hour of annealed steel. Large G.E.C. Batch and Continuous Furnaces have also been installed for processing in the light alloy, bronze and non-ferrous metal industries.

rely on the experience of

**G.E.C.**

GENERAL ELECTRIC COMPANY LTD., OF ENGLAND GENERAL ELECTRIC INC.



**Where sections  
vary greatly . . .**

**... 2 films are better than 1**

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The "sandwich" consists of two or more films of different speeds loaded into a standard cassette for simultaneous exposure. Thick sections of the specimen register clearly on the faster film, and the thinner sections

on the slower film. The effects of differential exposure can be accentuated by using lead screens in the "sandwich". The technique can also be used for making duplicate radiographs by using two films of the same speed.

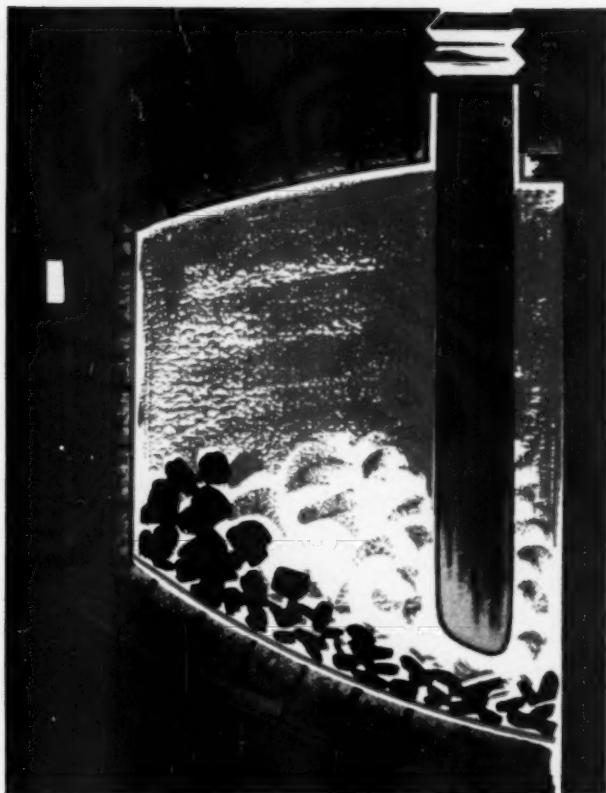
Choice of the appropriate ILFORD Industrial X-ray films obviously depends on the subject, the tube voltage and the presence or absence of screens. Full details of film choice and voltages, as well as lead screens, will be forwarded by request.

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**INDUSTRIAL X-RAY FILMS, CHEMICALS,  
CASSETTES AND LEAD SCREENS**

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"Acheson" Graphite Electrodes are manufactured from specially selected high grade raw materials which are carefully processed at all stages of manufacture under controlled conditions to give a final electrode for your arc furnaces with these properties at their optimum—

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**EXCEPTIONAL THERMAL SHOCK RESISTANCE**

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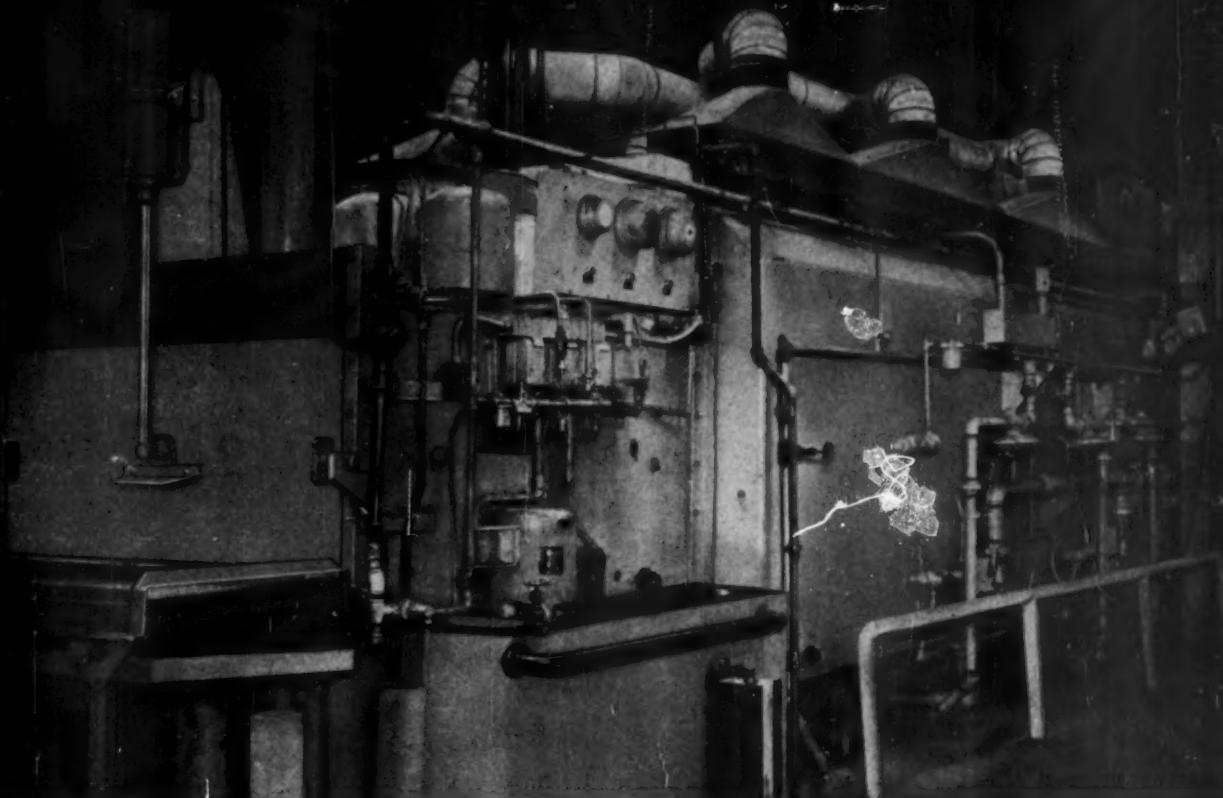
# Cronite

A GOOD NAME FOR NICKEL CHROME ALLOYS

**HEAT RESISTING  
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OF ALL TYPES**

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# Production Carburizing

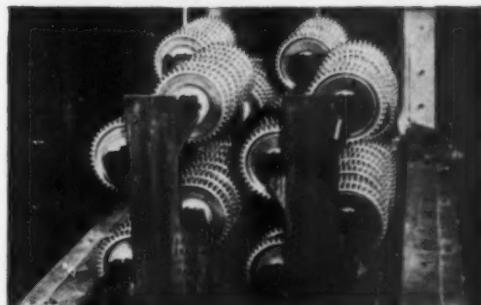
Five years ago the first Ipsen Double Row 4-Zone Pusher was built... see photo above. It is now six years since the first Deep Case Carburizing Unit was built. BIG Ipsen controlled atmosphere pushers have given unexcelled metallurgical results!

Check these features when you consider a carburizing furnace. They are exclusive with Ipsen.

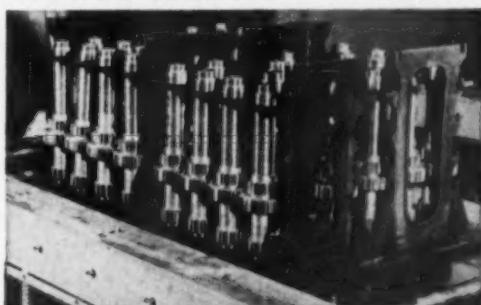
- Enclosed Charging Mechanism (Pat. No. 2,896,775)
- Chain Discharging Mechanism (Pat. No. 2,842,349)
- Baffles for 100% Forced Convection Heating (Pat. No. 2,788,205)
- Ceramic Heating Tube Mounting (Pat. No. 2,822,798)
- Ceramic "Flame-Busters" (Pat. No. 2,861,596)
- Ceramic Fan (Pat. No. 2,730,352)
- Water-Cooled Fan Motor (Pat. No. 2,800,317)
- Quench Oil Flow System (Pat. No. 2,854,013)

Other Patents Pending

Ipsen Pusher furnaces are described in Bulletin P-59. Send for your copy today.



This charge of steel gears is typical of work handled in Ipsen Production Carburizing Furnaces. Heating was 1700° F for 10 hours; oil quenched at 150° F. Case depth: .045".

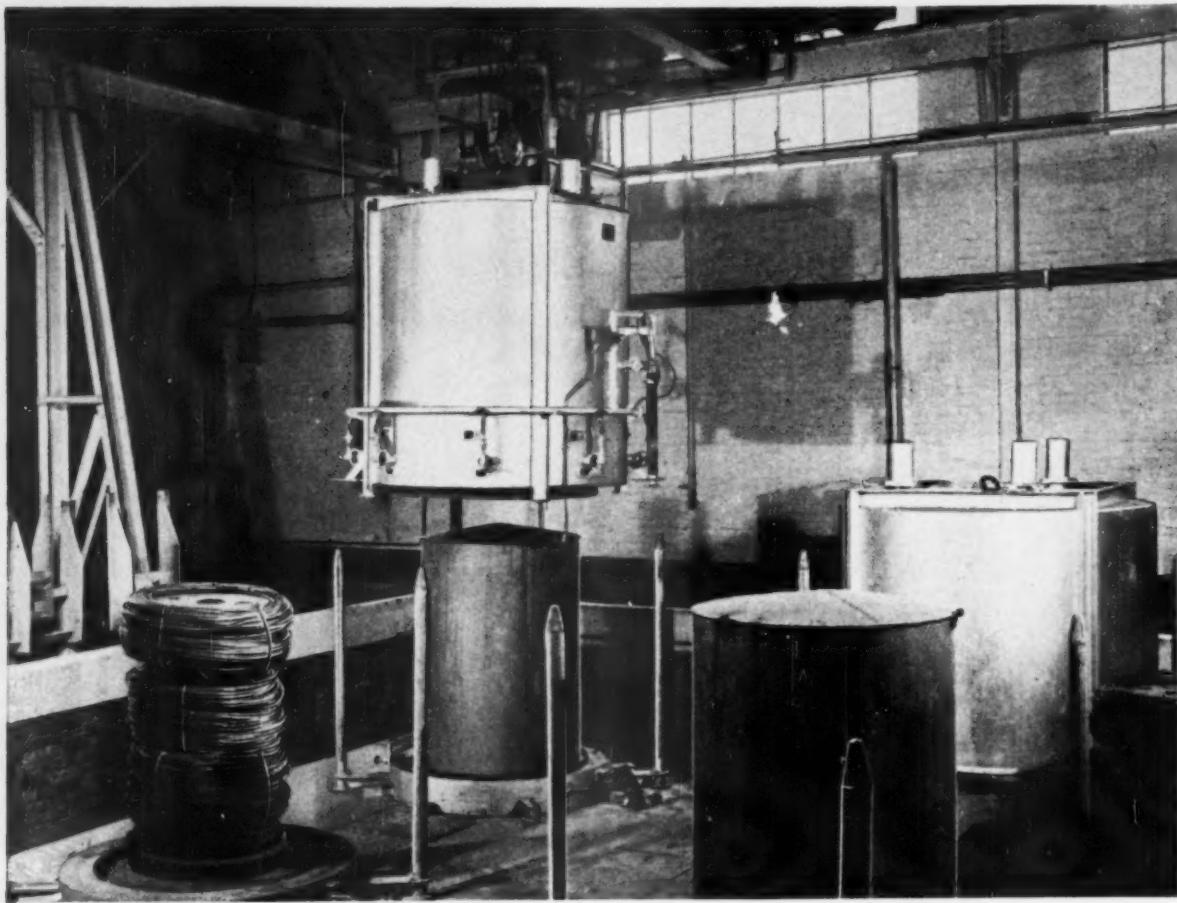


Countershafts ready for charging into Ipsen Production Carburizing Furnace. Heating time was 10 hours at 1700° F; quenching was in oil at 150° F. Case depth: .045".



4-0

IPSEN INDUSTRIES, INC., 153 VICTORIA ROAD, SURBITON, SURREY, Phone: ELMbridge 2021

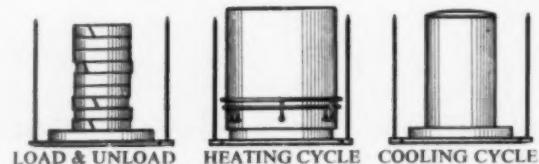


## Availability plus!

Incandescent lift-off furnaces work at full stretch *all the time*. No wasted downtime while waiting for a charge to be loaded or unloaded: no sooner is the heating cycle complete than the heated furnace is transferred to the next base. Meanwhile the first charge cools - under atmosphere if required - and is unloaded by the crane. Every heating cycle can be tailored exactly to meet the most stringent requirements, with full control over heating rates.

Incandescent lift-off furnaces are in use the world over, for ferrous and non-ferrous wire, rod, sheet and strip, and for the heat treatment of castings.

*With just one furnace  
several bases are  
in full use—*



# INCANDESCENT

FOR ALL HEAT TREATMENT PLANT

THE INCANDESCENT HEAT CO. LTD. SMETHWICK, ENGLAND

13/10D/60

# B-C-R copper

wire bars

ingot bars

billets

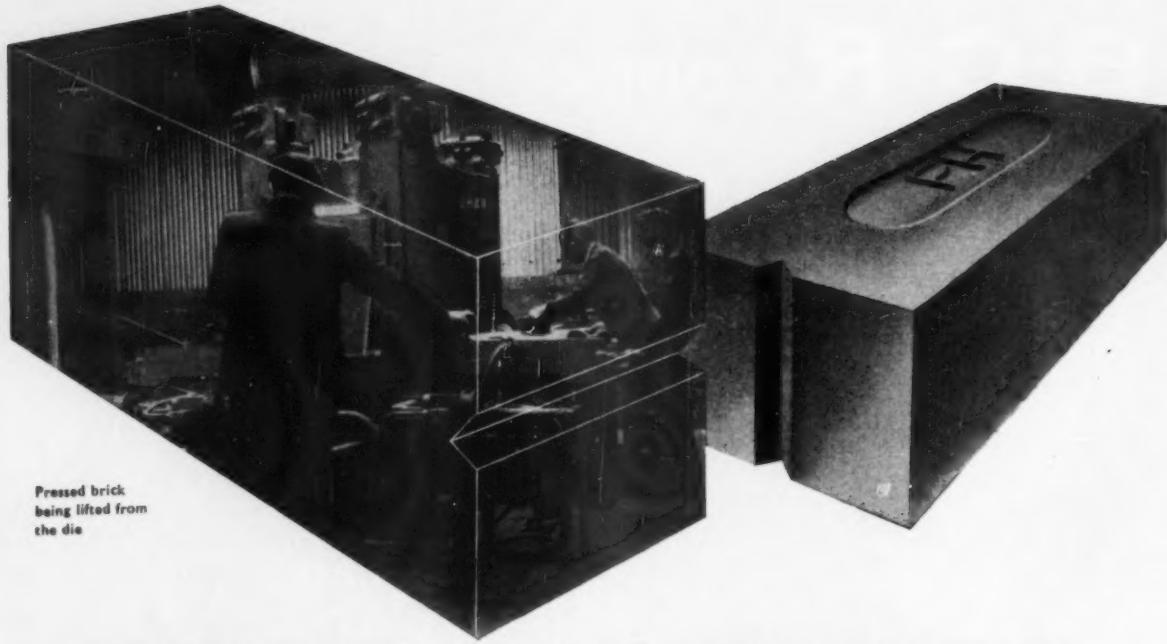
cakes

*Pouring 300 mm.  
Tough Pitch Billets.*



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More and more Pickford Holland refractory bricks are being supplied to steel and other industries throughout the world and the demand still grows. This surely points to the success of this policy of plant modernisation and is a tribute to the lasting service that these bricks give.



**PICKFORD HOLLAND** *Refractory Bricks*

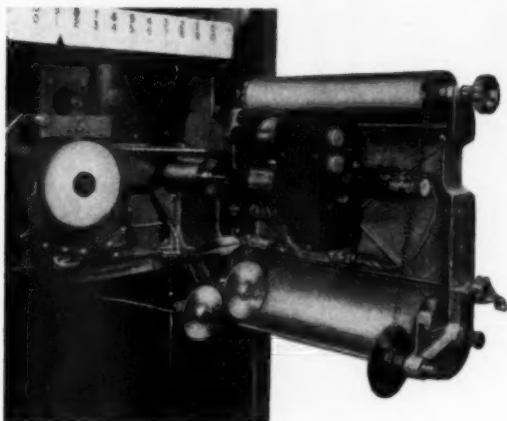
Consistent in Size, Shape, Texture and Performance

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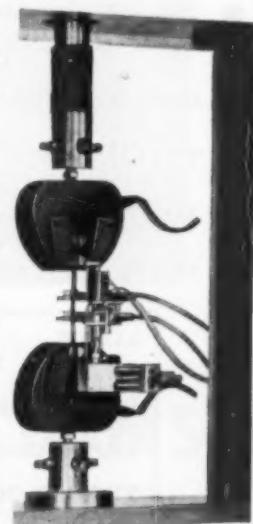
**The XY Chart Drive Accessory—what it is :**  
a high performance servo system for driving the chart of the Instron recorder. Operable from most types of extensometers, including differential transformers, small synchros, resistance potentiometers and strain gauges.

Varying degrees of magnification, depending on extensometer type and gear ratio for everything from metals to miracle plastics.

Unlimited chart travel to enable switching from extensometer to time drive for relaxation measurement.

This accessory performs equally well with all Instrons, no matter what their vintage. Installation is no problem.

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## THE NEW ALPHA DUROMETER SEOMATIC

FOR AUTOMATIC CHECKING OF ROCKWELL  
HARDNESS

SEOMATIC IS THE OUTCOME OF 60  
YEARS' EXPERIENCE IN THE FIELD OF  
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| OVERALL HEIGHT ... | 28.5 in. |
| WIDTH ...          | 8.3 in.  |
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| NETT WEIGHT ...    | 198 lb.  |

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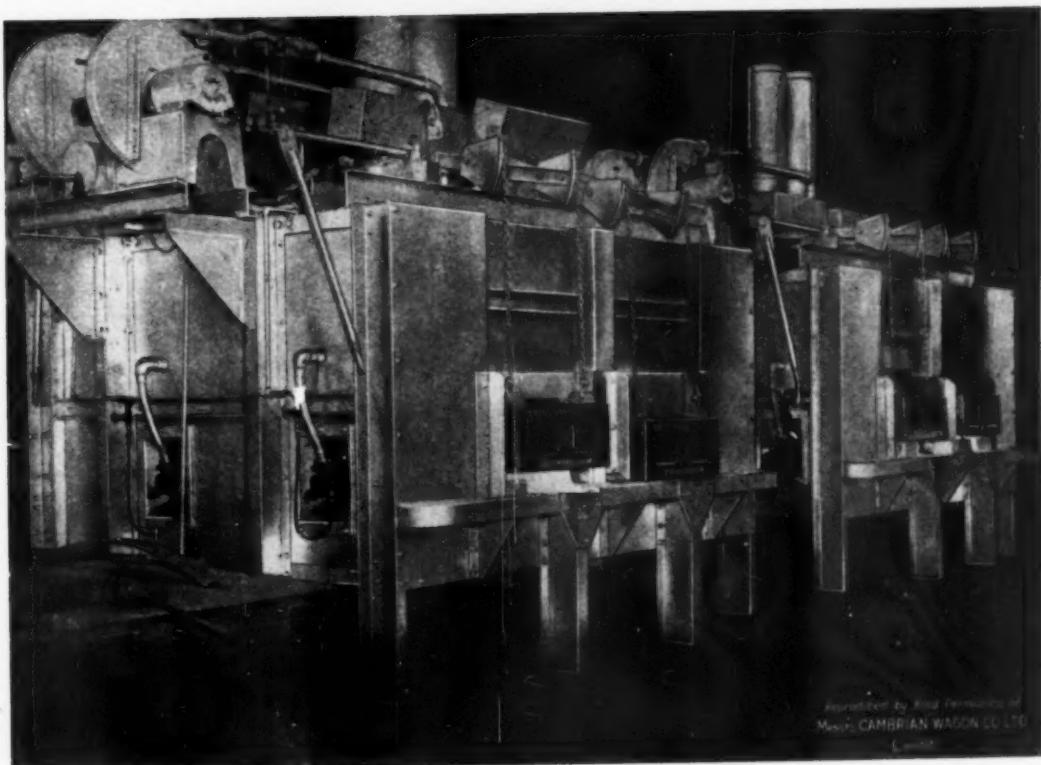
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Get the name right  
for controlled  
gas carburising . . .

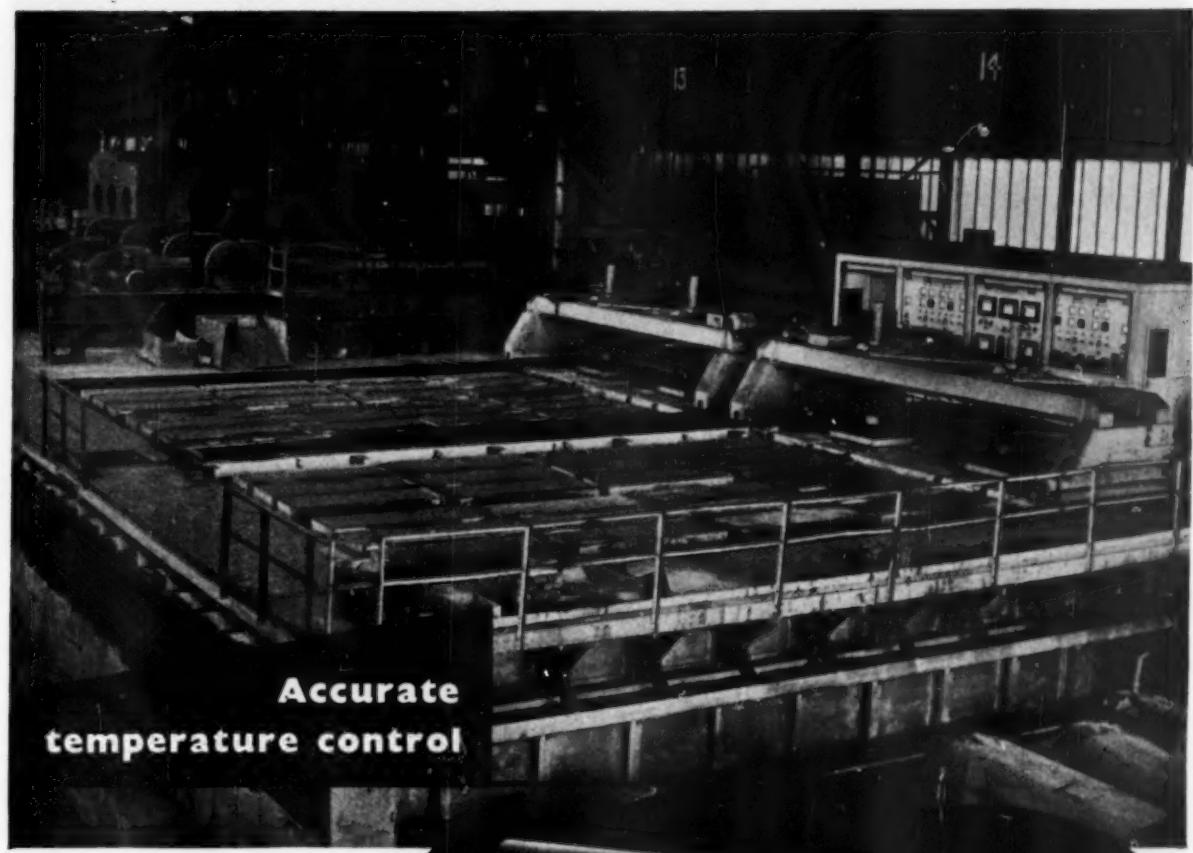
Minimum carburising time, fastest production rates and *full quality control*—these are the advantages of the Wild-Barfield Generated Gas and 'Carbodrip' methods. The benefit of many years' research is at your disposal when you write to us for advice on how gas carburising can give you a better product more efficiently.

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BARFIELD**



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W.B. 101



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temperature control**

The measurement and control of high temperatures is of fundamental importance in the manufacture of high quality steels, and platinum and platinum-rhodium alloy thermocouples have become the accepted method of exercising such control at all stages in the making of fine steels.

The manufacture of precious metal thermocouples demands raw materials of the highest purity together with strict scientific control at every stage of processing and testing. Thermocouples manufactured by the Baker Platinum Division of Engelhard Industries Ltd., have been proved to be of the highest possible quality, conforming in every respect to the relevant requirements of B.S.1826:1952 and B.S.1041:1943.

Continued research by Engelhard Industries in the field of high temperature pyrometry has resulted in two recent developments:

**THE INTEGRAL-SHEATH THERMOCOUPLE**, which has a diameter in the order of  $\frac{1}{8}$  in. and combines compactness, flexibility and resistance to thermal shock to an extent unattainable with conventional sheathed assemblies.

**'FIBRO' THERMOELEMENT PLATINUM**, which maintains high strength and ductility after long periods at high temperatures whilst retaining all the thermoelectric attributes of normal thermoelement quality platinum.

★Write for copy of new illustrated leaflet.



*A view of the electric soaking pits at the works of The Consett Iron Company which are controlled by means of 95% platinum-5% rhodium versus 80% platinum-20% rhodium thermocouples.*

**BAKER  
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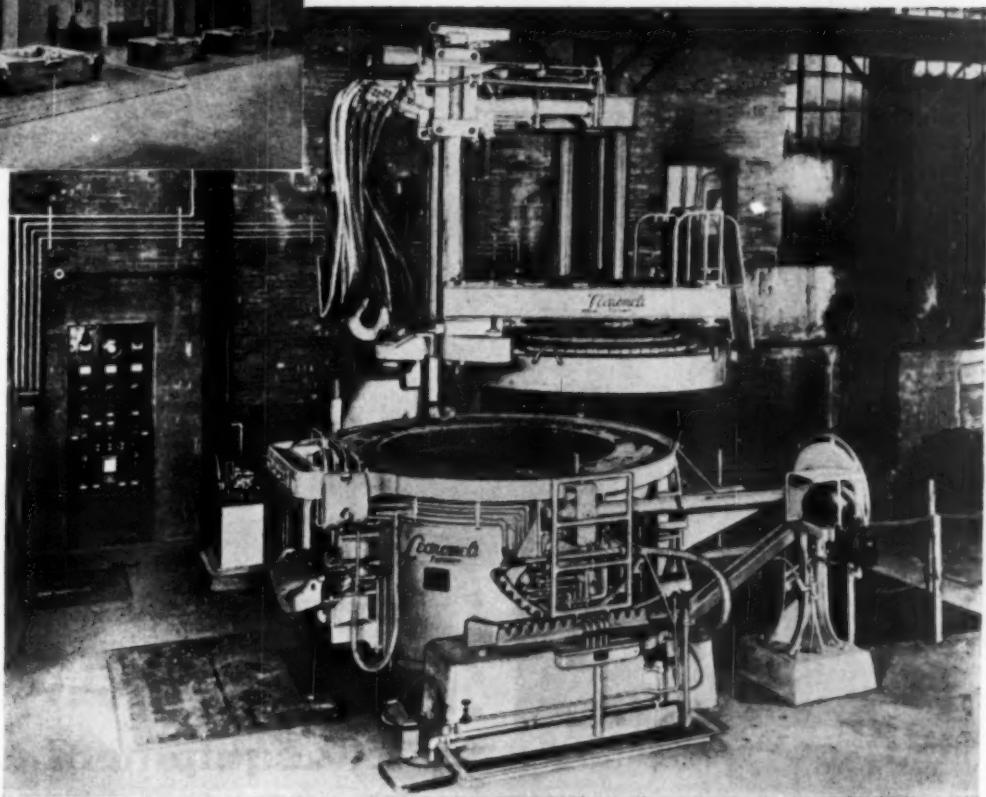
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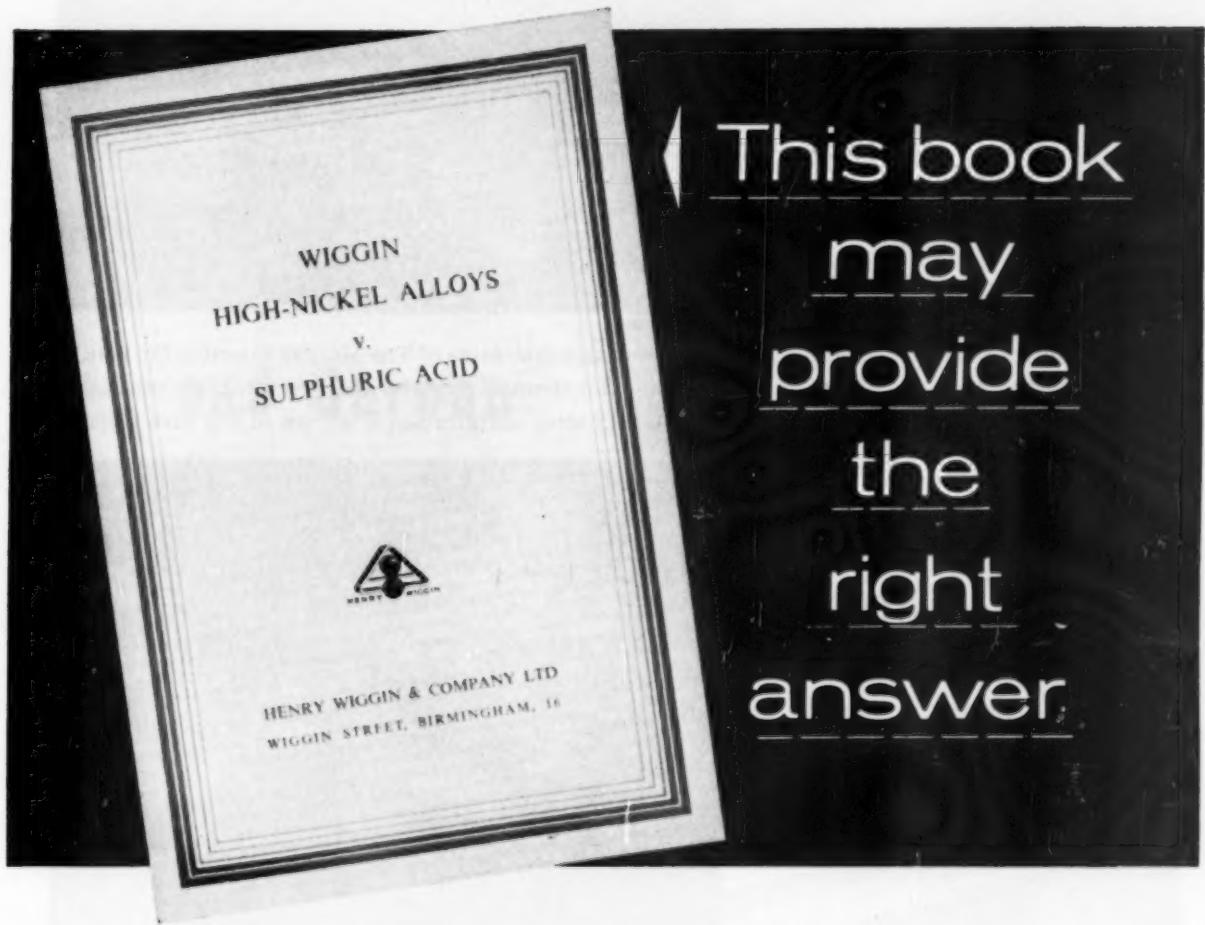


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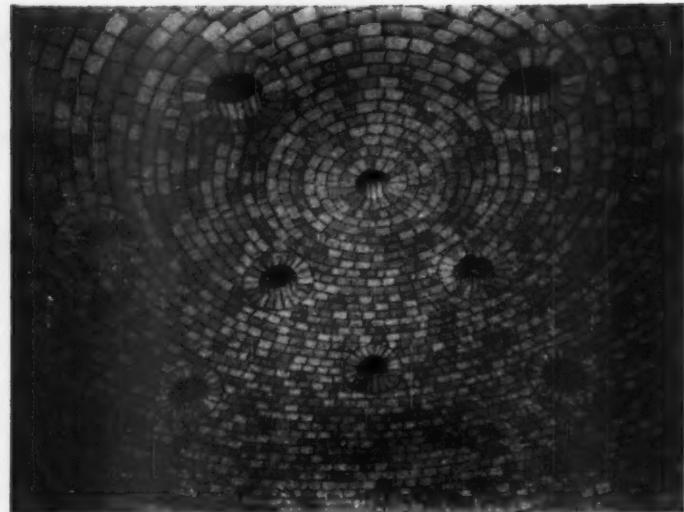
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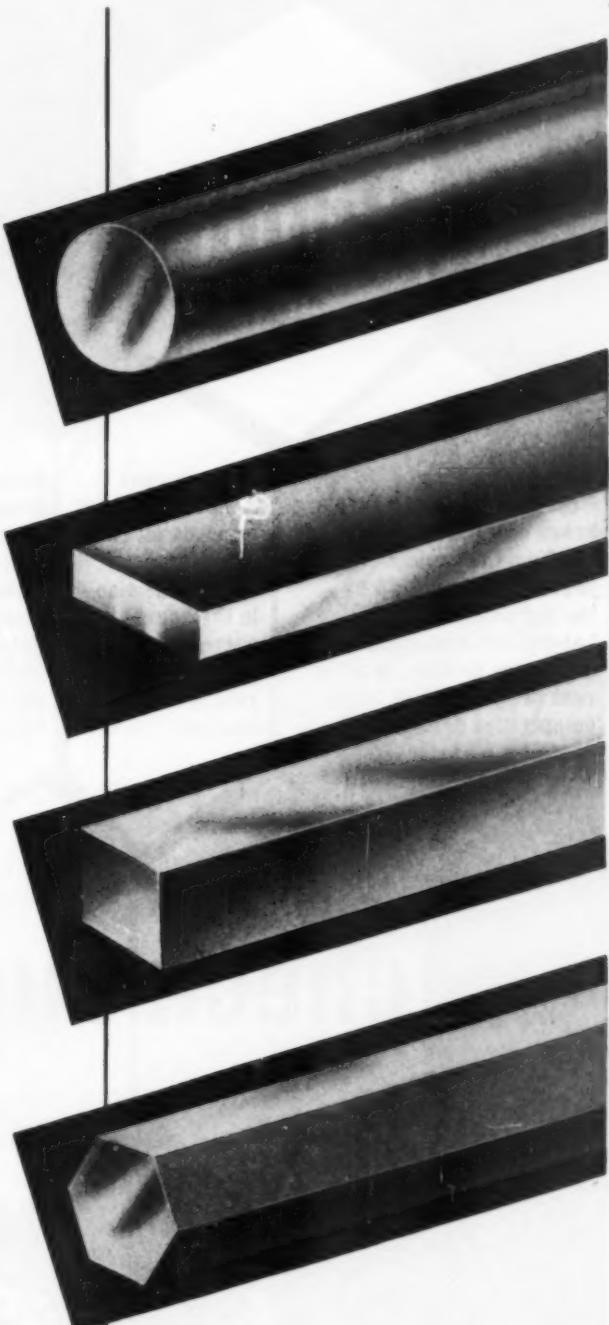
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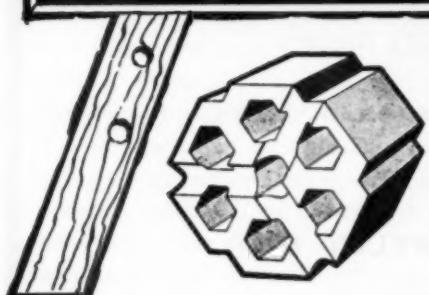
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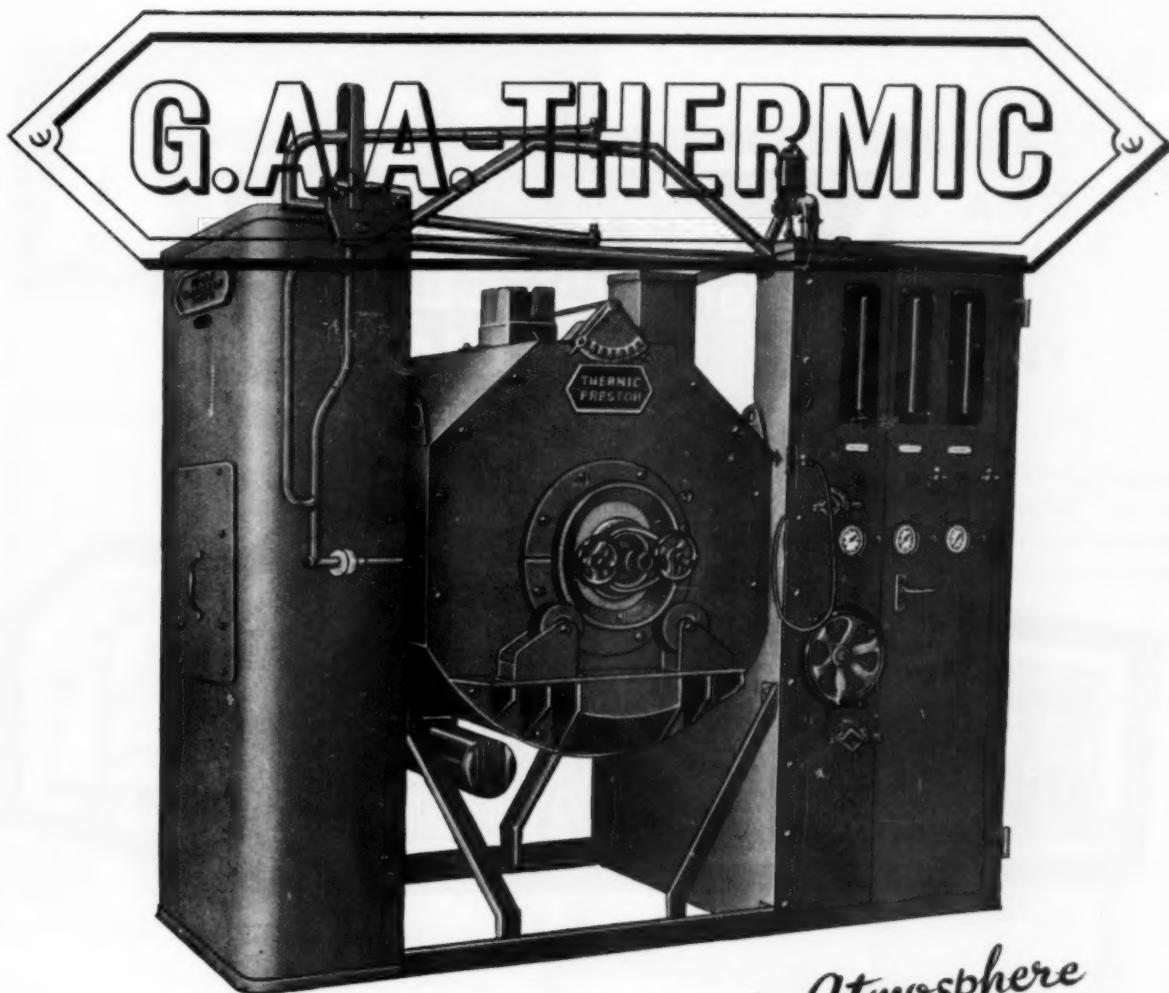
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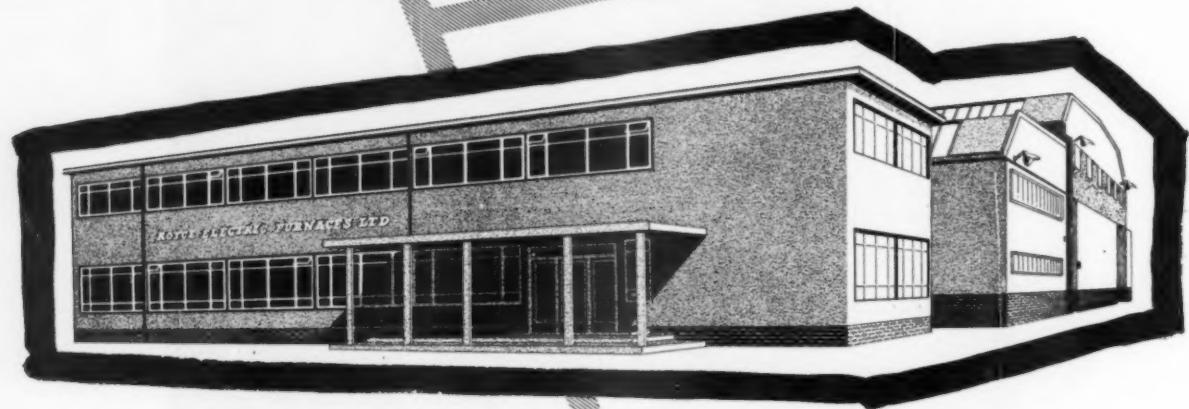
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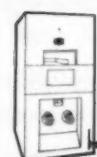
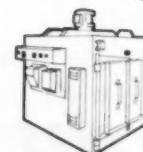
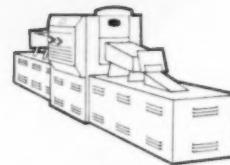


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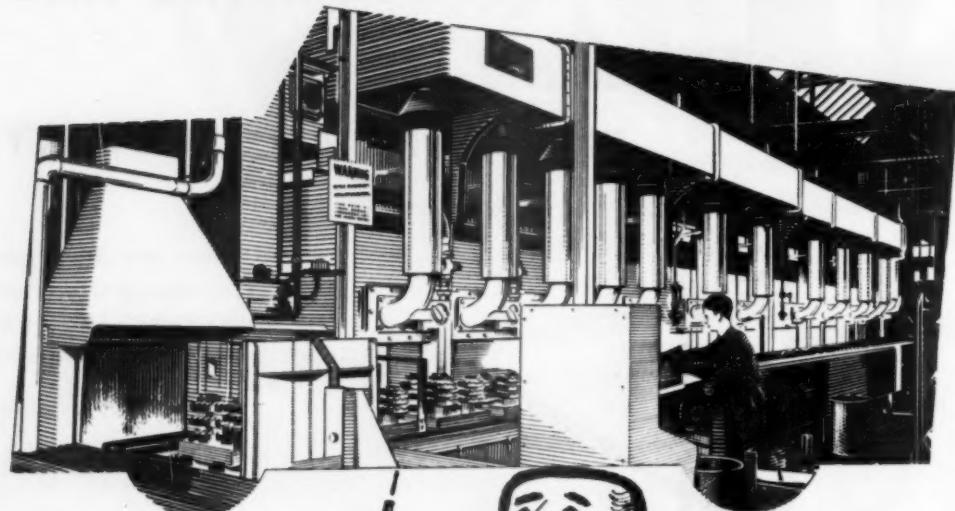
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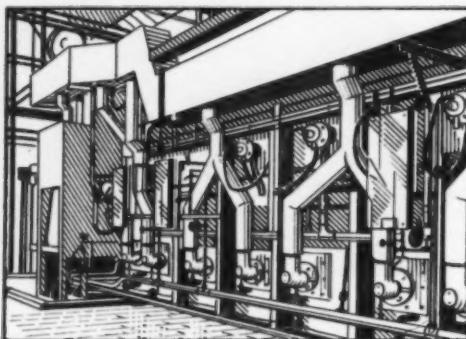
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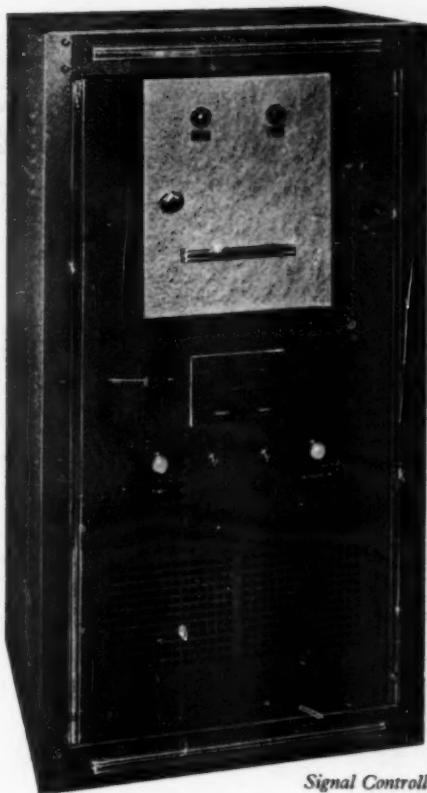
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INCORPORATING THE METALLURGICAL ENGINEER

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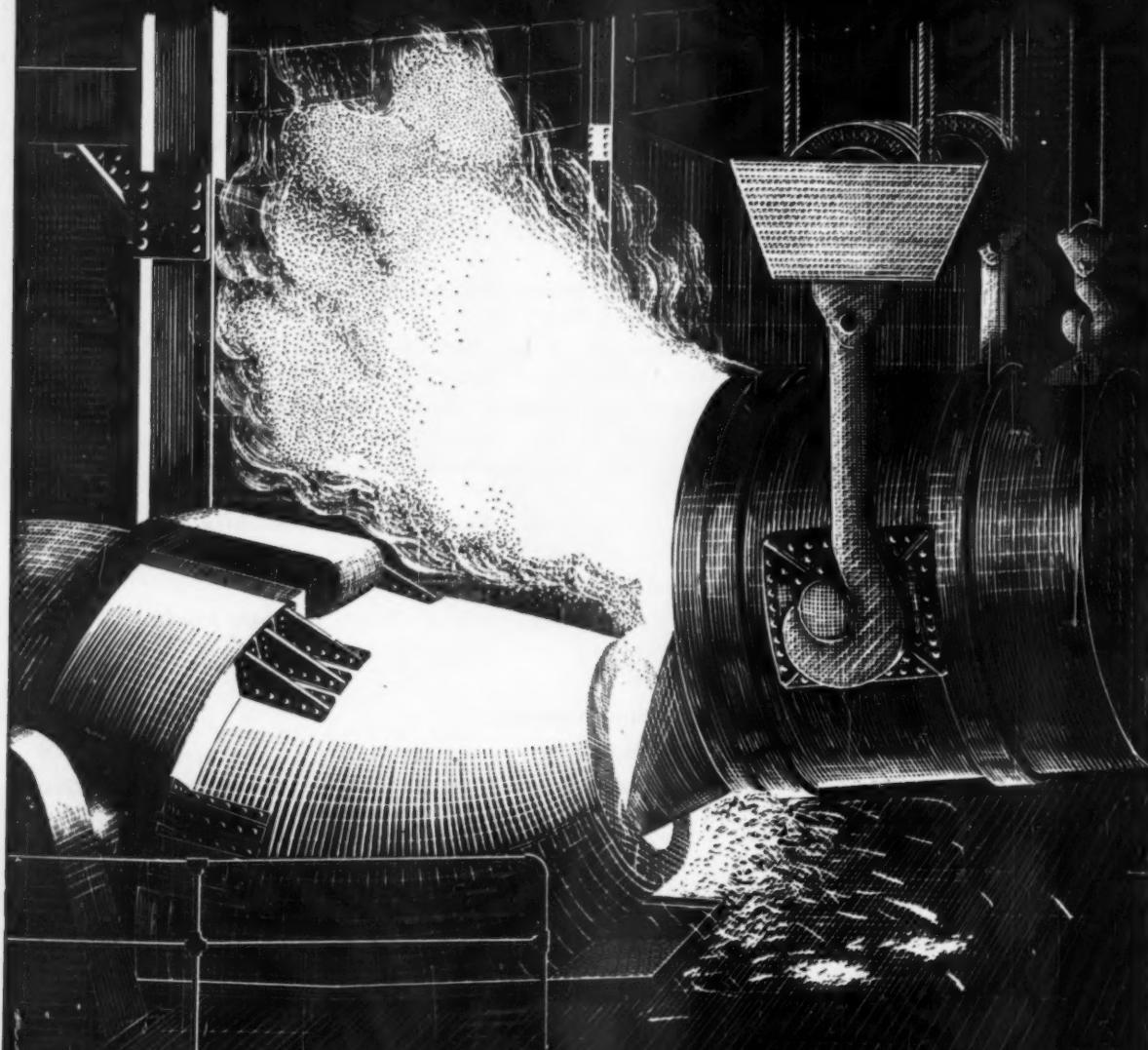
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# METALLURGIA

THE BRITISH JOURNAL OF METALS  
INCORPORATING THE "METALLURGICAL ENGINEER"

DECEMBER, 1960

Vol. LXII. No. 374

## Steelmaking: The Impact of Oxygen\*

THE advent of relatively cheap oxygen in bulk is revolutionising steelmaking throughout the world. The pattern of steelmaking has remained almost unchanged for many years, but the first ripples of a tremendous wave of change are now passing through the industry. The open hearth furnace process has been the traditional basis of the British industry, accounting for some 85% of all steel production, but development plans now being executed and in preparation show a sharp increase in the use of the new converter processes and of electric steelmaking methods. It is estimated that this will reduce the predominance of the open hearth furnace to about 70% of the potential steel production of 34 million tons in 1965. The potential production percentages for other processes are as follows, the actual production percentage for 1959 being given in parentheses: electric 12 (6½), Bessemer 6 (7), L.D. 11 (nil), Kaldo 2 (nil), others nil (½). In spite of this proportionate reduction, the tonnage of steel to be made in open hearth furnaces should nevertheless increase substantially.

The open hearth furnace offers great flexibility in the relative use of pig iron and scrap and in the qualities of steel it can produce, and gives close control of quality. The traditional open hearth furnace is, however, handicapped compared with the new converter processes using oxygen by its relatively slow operation and low hourly production rate. But the magic key to higher productivity, oxygen, is not limited in its application to converters. Many British companies already use, or are planning to use, oxygen in their open hearth furnaces, where dramatic increases in productivity are possible with the intensive use of oxygen.

For many decades steelmakers have realised that pure oxygen can revolutionise steelmaking by making possible the development of new techniques and by considerably speeding up reaction rates in the traditional processes. It was not until the early 1950's, however, that the use of high purity oxygen—"tonnage" oxygen—in bulk became an economic possibility. Since then the L.D., Kaldo and Rotor processes—consuming about 2,000 cu. ft. of oxygen per ton of steel—have been introduced. At the same time the air blast of the traditional basic Bessemer converter has been superseded at an increasing number of plants by a blast of oxygen-enriched air, or by a mixture of oxygen and steam—requiring 1,500/1,800 cu. ft. of oxygen per ton of steel. All the three basic Bessemer plants in the United Kingdom are already equipped to use oxygen and this technique is used for a large part of their production. The use of oxygen has also

enabled a number of the industry's open hearth furnaces to achieve notable increases in production rates, either, as at Brymbo, at Richard Thomas and Baldwins, Redbourn, and at Skinningrove, by using a molten iron which has been pre-refined as a result of oxygen blowing, or, as at The United Steel Companies' Appleby-Frodingham works, at Consett, and at The Steel Company of Wales, by the application of oxygen within the furnace itself. In the three last-named cases oxygen is used for enrichment of the combustion air, or as a jet to speed up production or assist in attaining the very low carbon levels required in steels to be subjected to deep-drawing. An outstanding example in the United Kingdom at the present time is the use of up to 2,500 cu. ft. of oxygen per ton of steel in the Ajax furnaces

at Appleby-Frodingham, which has increased production from 63% to 103% over the former rate, and at the same time reduced fuel consumption to one-fifth of its former level. More and more open hearth furnaces will use pre-refined metal or be themselves converted to oxygen use. The use of oxygen in open hearth furnaces invariably leads to a reduction in fuel consumption, although not necessarily to so spectacular an extent as is achieved in the Ajax furnace. When one considers as well the increased production from converters, which require no external supply of fuel, and the swing to electric arc furnaces, it seems that, although the total quantity of fuel used for steelmaking will continue to rise, the amount required per ton of steel will show an appreciable reduction from the present figures.

Comparing 1959 with 1958, the U.K. use of oxygen for steelmaking almost doubled, the largest proportionate increases being in its use for combustion air enrichment in open hearth furnaces and in the various converter processes, the latter increase being sevenfold. These increases are only a beginning, for a new converter steelmaking installation is already in operation at Richard Thomas and Baldwins' Ebbw Vale works. Others are coming into operation at Colvilles' Ravenscraig Works, at Consett, at John Lysaghts' Scunthorpe works and at Richard Thomas and Baldwins' new Spencer works at Newport. Further major converter steelmaking developments are in an advanced stage of preparation. These, together with increased consumption in open hearth and electric furnaces, will mean that the amount of oxygen used for pre-refining and steelmaking will rise by 1965 to possibly eight times the 1959 level of consumption. Oxygen is widely used in steelworks for many purposes besides steelmaking, particularly for the preparation of ingot, slab and billet surfaces prior to rolling, for general engineering and for scrap preparation. By 1965 these non-metallurgical uses may require a total of 2½ to 3 times their 1959 level of oxygen consumption.

In blast furnaces, too, oxygen may have an important part to play. Here it can be used, if necessary, in a relatively low purity condition to raise the oxygen con-

\* Reprinted with slight modification from *Steel Review* No. 20, October, 1960.

tent of the blast for the furnaces from the normal 21% to 25%. The main effect of such enrichment is to increase the quantity of iron produced per day. The results of tests made by The Steel Company of Wales have already led them to order a tonnage oxygen plant to supply 630 tons of 90% purity oxygen per day for use in three of its blast furnaces. A number of other companies too are planning to use oxygen enrichment of the blast. Since oxygen plant with a capacity of 100 tons per day is needed to enrich the air blast of a blast furnace producing 1,000 tons of iron per day to a level of 25% oxygen, there are considerable possibilities for the increased use of oxygen in ironmaking.

The industry already has eight high purity tonnage oxygen plants in operation with a daily capacity of

1,125 tons. Orders have been placed for additional plants with a capacity of 1,775 tons per day of high purity oxygen and 630 tons per day of 90% purity oxygen, all of which are expected to be in operation by the middle of 1962. Negotiations for further plants are at present being conducted. Many companies are too small to justify the construction of individual tonnage plants and not near enough to other oxygen users to enable them to use a common plant and ring-main delivery system, and they will continue to receive their oxygen in liquid form from road tankers, evaporating it as required. One can reasonably forecast that by the mid 1960's the total oxygen consumption of the steel industry for all purposes will rise to 5,000 tons per day, or 1.8 million tons per year.

## Meeting Diary

3rd January

**Institute of Metals, Oxford Local Section.** "Meteorites," by H. J. AXON. Cadena Café, Cornmarket Street, Oxford. 7.15 p.m.

4th January

**Leeds Metallurgical Society.** "Some Recent Developments in the Welding of Metals," by E. N. GREGORY. University Staff House, University Road, Leeds. 6.30 p.m.

**Society of Chemical Industry, Corrosion Group (Cathodic Protection Panel Meeting).** Discussion on Galvanic Anodes. Society headquarters at 14 Belgrave Square, London, S.W.1. 6 p.m.

5th January

**Institute of Metals, Birmingham Local Section.** "Vacuum Technology," by T. T. THORLEY. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

**Institute of Metals, London Local Section.** "Metallurgy in Electronics," by J. E. HUGHES. Institute headquarters, 17, Belgrave Square, London, S.W.1. 6.30 p.m.

9th January

**Institute of British Foundrymen, Sheffield Branch.** "Pattern Equipment for Shell Moulding," by E. C. ROWEN. Technical College, Pond Street, Sheffield. 7 p.m.

10th January

**Institute of British Foundrymen, London Branch (Slough Section).** "Coremaking—Modern Methods and Machinery," a Brains Trust with J. FALLOWS, J. HARVEY and R. SUTCLIFFE on the panel. Lecture Theatre, High Duty Alloys, Ltd., Slough. 7.30 p.m.

11th January

**Institute of British Foundrymen, London Branch (Southampton Section).** Technical film show: "The Making, Structure, Heat Treatment and Properties of Steel." Technical College, St. Mary's Street, Southampton. 7.30 p.m.

**Manchester Metallurgical Society.** "Modern Methods of Metallurgical Analysis," by K. M. BILLS. Manchester Literary and Philosophical Society, George Street, Manchester. 6.30 p.m.

12th January

**East Midlands Metallurgical Society.** "The Metallurgy of Semi-Conductors," by J. G. WILKES. Derby and District College of Art, Derby. 7.30 p.m.

16th January

**Institute of British Foundrymen, London Branch (East Anglian Section).** "Origin, Detection and Elimination of Gases in Cast Metals," by D. V. ATTERTON. Lecture Hall, Public Library, Ipswich. 7.30 p.m.

17th January

**Institute of Metals, South Wales Local Section.** "Metals and Alloys Used in the Jewellery and Silverware Industry," by Miss D. L. PILE. Royal Institution, Swansea. 7 p.m.

18th January

**Institution of Plant Engineers, Kent Branch.** "Photography in Industry," by N. E. SHERLOCK. King's Head Hotel, High Street, Rochester. 7 p.m.

**Society of Chemical Industry, Corrosion Group.** "Metallic Diffusion Coatings," by R. L. SAMUEL. Society headquarters, 14, Belgrave Square, London, S.W.1. 6 p.m.

18th-19th January

**Institution of Mechanical Engineers.** Symposium on Pressure Vessel Research Towards Better Design.

19th January

**Institute of Metals, Birmingham Local Section.** "The Heat Treatment of Engineering Components," by E. MITCHELL. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

**Institute of Metals, Sheffield Local Section.** "Ductile Fracture," by Professor R. W. K. HONEYCOMBE. Applied Science Building, The University, St. George's Square, Sheffield. 7.30 p.m.

**North East Metallurgical Society.** "The Effects of Nuclear Radiation on Metals," by H. M. FINNISTON. Cleveland Scientific and Technical Institution, Corporation Road, Middlesbrough. 7.30 p.m.

25th January

**Institute of British Foundrymen, London Branch.** "Developments in the Production of Gunmetal Castings," by F. HUDSON. Constitutional Club, Northumberland Avenue, London, W.C.2. 7.30 p.m.

**Manchester Metallurgical Society.** "The X-Ray Micro-analyser as a Research Tool for the Metallurgist," by D. A. MELFORD. Manchester Literary and Philosophical Society, George Street, Manchester. 6.30 p.m.

26th January

**Southampton Metallurgical Society.** "Hot Dip Galvanising," by M. H. DAVIS. Southampton University. 7.15 p.m.

**West of England Metallurgical Society.** "Recent Developments in Metallography," by Professor R. W. K. HONEYCOMBE. College of Technology, Ashley Down, Bristol. 7.30 p.m.

27th January

**North East Coast Institution of Engineers and Ship-builders.** "Fatigue Strength of Marine Shafting," by G. P. SMEDLEY and B. K. BATTEN. Mining Institute, Newcastle upon Tyne. 6.15 p.m.

31st January

**Sheffield Metallurgical Association.** "Tool Steels," by G. HOYLE. B.I.S.R.A. Laboratories, Hoyle Street, Sheffield. 7 p.m.

UNDER a suggestion scheme run by The Morgan Crucible Co., Ltd., Battersea, Mr. J. H. Fairweather, laboratory assistant in the company's research department, has been awarded £225. His alteration to the cooling system of a laboratory furnace will save approximately 10 million gallons of water a year, and represents an annual saving to the company of £400.

A CONTRACT worth £339,000, for six steelworks process line drives, has been received by Associated Electrical Industries, Ltd., from Richard Thomas and Baldwins, Ltd., for installation at the new Spencer Works at Llanwern, near Newport, Monmouthshire. All the line drives are to be completely erected, commissioned and available for commercial production by the end of September, 1961.

# Physical Properties and Constitution of Liquid Slags

By B. T. Bradbury,\* B.Met., Ph.D., and D. J. Williams,† B.Sc., Ph.D.

*A knowledge of the structure of liquid slags is of vital importance in leading to a better understanding of their functions in process metallurgy. Interest in their structure has grown with the development of the thermodynamic approach to slag/metal reactions, and in many instances a structural model is an important aid to the correct interpretation of these reactions. In the present article the authors review the physical properties of simple slag systems, paying particular attention to the more recent findings in this field of metallurgical research.*

THE constitution of liquid silicates, phosphates and borates has been studied extensively in the past.

Many physical measurements have been made to determine the structure of these melts, and to aid in the control of technological processes. Yet in spite of the more recent work which has been carried out, particularly by Bockris and his co-workers, there does not seem to have been a review article in this field of research in a British journal for some time.

In the past, many of the investigations of physical properties have been concerned with more complex melts closely related in composition to the slags used in steel and glass-making processes. It seems advisable however to interpret the physical properties of simpler binary melts initially, before considering the complex technologically important systems.

Physical properties such as electrical conductivity, viscosity, density, and to some extent, surface tension, have played the most important role in the elucidation of the structure of silicate, phosphate and borate melts. More recently, additional information concerning structural changes occurring within these complex melts has been obtained from infra-red absorption measurements made on quenched samples obtained from the melts.

## I. SILICATE SYSTEMS

There is a considerable number of papers<sup>1-5</sup> in the literature concerned with the molten silicates, but since most of the data recorded have been remeasured and considerably supplemented by Bockris *et al.*, and by Shartsis and Spinner, only the more recent work will be described here.

### Electrical Conductivity Measurements

Attention has been focused upon electrical properties, particularly electrical conductivity, by many workers in the past. Such measurements offer the possibility of obtaining a measure of the inter-ionic forces in liquid slags. Hence, information concerning the freedom of movement of particles in the network lattice and the general type of structure existing can be obtained. Bockris<sup>6</sup> and co-workers have studied the electrical properties of a series of binary silicates. Melts were contained in a molybdenum crucible and molybdenum electrodes were employed to minimise contamination.

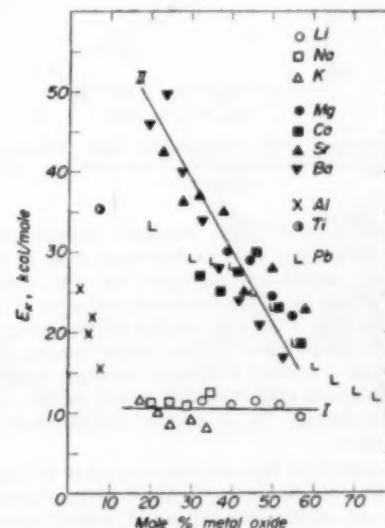


Fig. 1.—Variation with composition and differing cations of the energy of activation of electrical conductivity ( $E_a$ ) for binary silicates.<sup>41</sup>

In the case of those systems exhibiting low conductivity values, where vacuum studies were necessary, all-glass vacuum cells were constructed for use at temperatures up to 2,300° C. The results which were obtained indicated the predominantly ionic nature of silicate melts, as demonstrated by the following evidence:—

- the electrical conductivity is of the same order of magnitude as for ionic melts, e.g. fused salts;
- the ratio of the conductance above and below the liquidus temperature is about 100;
- the temperature coefficient of conductance is positive; and
- the passage of current through the melt produces electrolysis.

In all cases, the Rasch-Hinrichsen Law is obeyed:—

$$K = A_E \exp. (-E_a/RT) \quad (1)$$

where  $K$  is the specific conductance,  $A_E$  is a constant,  $E_a$  is the energy of activation for conductance,  $R$  is the

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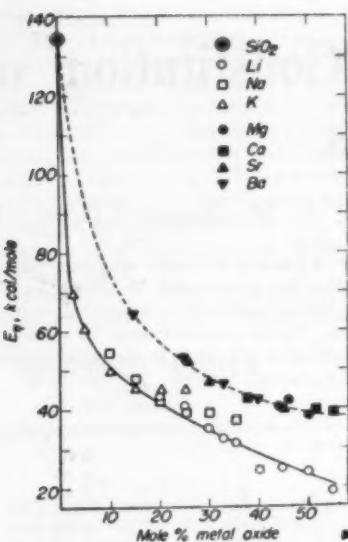


Fig. 2.—Variation with composition and differing cation of the energy of activation of viscous flow ( $E_a$ ) for binary silicates.<sup>41</sup>

gas constant and  $T$  is the absolute temperature ( $^{\circ}\text{K}$ ). Thus, by measuring the conductance over a range of temperature, activation energies for the conduction process ( $E_a$ ) can be evaluated from equation (1). These are shown in Fig. 1 for the various silicate systems as a function of composition. From these results, Bockris<sup>6</sup> *et al* observed a marked difference between systems containing alkali and alkaline earth metal oxides, although within each group,  $E_a$  is nearly independent of the cationic species.

It is evident that the cationic charge is an important factor in the mechanism of electrical conduction. In those systems containing Group I metal cations,  $E_a$  seems to be independent of composition, but in the case of Group II cations, it increases with decreasing metal oxide content.

Bockris<sup>6</sup> has developed a satisfactory interpretation of these results by assuming that the addition of metal oxide to silica results in the breakdown of the three-dimensional silica network into silicate anions, and by correlating the free energy of activation for conductance with the "ion-oxygen attraction parameter." This parameter, which is taken to be a measure of the electrical interaction between the metal cation and the silica network, is defined algebraically as  $I = Ze^2/R$ , where  $Z$  is the valence of the cation,  $e$  is the electronic charge, and  $R$  is the ionic radius of the cation.

Thus,  $I$  values are small for Group I cations, and since conductivity is a cationic process,  $E_a$  values will be small in these cases. The addition of more alkali metal oxide has very little effect upon  $E_a$  values because of the small  $I$  values. For the alkaline earth ions, the effect is different due to their larger  $I$  values. Energies of activation are high with small amounts of metal oxide, because the cations have high energy barriers to surmount under the influence of an electric potential. Further additions of metal oxide break up the silica lattice, and the cation moves more freely under the influence of an electric field:  $E_a$  values are thus reduced.

### Transport Number Measurements

Further work by Bockris<sup>7</sup> and co-workers has confirmed the idea of the ionic nature of silicates as indicated by the electrical conductivity work. Faraday's Laws of electrolysis were checked for the systems  $\text{Li}_2\text{O-SiO}_2$ ,  $\text{K}_2\text{O-SiO}_2$ ,  $\text{MnO-SiO}_2$ , and  $\text{PbO-SiO}_2$ , and transport numbers were determined in the lithium and potassium silicates.

The check on Faraday's Laws is based essentially on the fact that the electrolysis of a silicate involves the deposition of metal at the cathode and evolution of oxygen at the anode. Faraday's Laws may be tested by measuring either one or the other. There is some difficulty in estimating the amount of metal cathodically deposited, and the method usually adopted is to employ a graphite anode with which the evolved oxygen reacts. The volumes of  $\text{CO}$  and  $\text{CO}_2$  resulting from the reaction may then be used to check the laws of electrolysis.

A vacuum furnace connected to a gas analysis line was employed for the experiments, and the melt was contained in a molybdenum crucible. The current passed through a molybdenum cathode and a graphite anode. The results obtained vary with the binary system under investigation. In the case of  $\text{Li}_2\text{O-SiO}_2$  melts of varying compositions, the passage of 1 Faraday of electricity caused the evolution of 0.98 g. equiv. (gram equivalent) of oxygen. Although the results for this system indicate complete ionic conduction as do the results for  $\text{PbO-SiO}_2$  melts, in the case of the  $\text{CaO-SiO}_2$  and  $\text{MnO-SiO}_2$  systems, certain side reactions take place which give rise to spurious results. For the  $\text{CaO-SiO}_2$  system, with metal oxide content from 47% to 55%, the evolution of oxygen is 1 g. equiv. per Faraday of electricity, but as the  $\text{SiO}_2$  content is increased the amount of oxygen evolved exceeds 1 g. equiv. per Faraday. This excess evolution has been interpreted as being due to the side reaction:—



$\text{SiC}$  was detected by X-ray techniques. For the  $\text{MnO-SiO}_2$  system, however, less than 1 g. equiv. of oxygen is obtained for the passage of 1 Faraday, and it has been suggested that a side reaction occurs involving the production of metallic manganese. The metal is then oxidised to  $\text{Mn}^{2+}$ , the oxidation process reducing the efficiency of oxygen evolution.

Transport numbers were determined for melts in the systems  $\text{K}_2\text{O-SiO}_2$  and  $\text{Li}_2\text{O-SiO}_2$ , using an alumina cell fabricated with two electrode compartments and joined by an alumina bridge. Using a molybdenum rod for cathode and a graphite rod as anode, a known current was passed for a given period, and the change in composition in the anode and cathode compartments was estimated by chemical analysis.  $\text{K}_2\text{O-SiO}_2$  melts conduct completely cationically over the composition range studied (50-20 mole %  $\text{K}_2\text{O}$ ). The data for the  $\text{Li}_2\text{O-SiO}_2$  melts is by no means certain, since these melts attacked the alumina container cells. An approximate transport number of 0.8 for the  $\text{Li}^+$  ion was obtained.

### Viscosity Measurements

Whilst conductance properties are concerned with the cationic entities in the melt, viscosity measurements have given an insight into the structure of the silicate lattice. This has been achieved by the evaluation of both the energy of activation for the process, and its relation

to the size of the flow units present. Shartsis and Spinner<sup>8</sup> and Bockris<sup>9a,b</sup> and co-workers have measured viscosities in binary silicates. Bockris' interpretation of viscous properties seems to be the most developed and is described below.

Bockris and Lowe<sup>9a,b</sup> have measured the viscosity of a large number of binary silicate melts using a rotating crucible method. The crucible was equipped with a fixed centre spindle, and the torque on this spindle due to crucible rotation was measured, using an electromagnetic device. The damping effect of the melt on these rotational movements was related to the viscosity.

Viscosities were measured over a temperature range and, as with the electrical conductivity studies, an energy of activation for the process was determined using the empirical relation:

$$\eta = A_\eta \exp. (-E_\eta/RT) \quad (2)$$

where  $\eta$  is the viscosity,  $A_\eta$  is a constant,  $E_\eta$  is the energy of activation of the viscous process,  $R$  is the gas constant and  $T$  is the temperature (°K). The results which have been obtained by Bockris and Lowe, and Shartsis and Spinner for the binary silicates are summarised in Fig. 2.

The sudden drop in the energy of activation for viscous flow from 140 k. cal./mole to around 50 k. cal./mole on the addition of 10 mole-% metal oxide is assumed to indicate a radical structure breakdown. Since in any liquid the viscous drag between the layers of moving liquid will be greater the greater the size of the molecules or ions present, a reduction in the energy of activation for viscous flow in the silicates can be taken as an indication of a reduction in the size of the silicate anion units.

A particularly interesting feature of Bockris' results is the continuous and gradual fall in  $E_\eta$  values as the proportion of metal oxide increases beyond 10 mole-%. This observation is compatible with a gradual breakdown of the silicate structure and invalidates the theory put forward by Endell and Hellbrügge<sup>10</sup> in 1942. These workers interpreted the plots shown in Fig. 2 on the basis of a gradual build up of infinite silicate chains as the proportion of metal oxide decreased. At the orthosilicate composition ( $\text{SiO}_4$ )<sup>4-</sup> ions were believed to be present in the melt, and on addition of  $\text{SiO}_2$

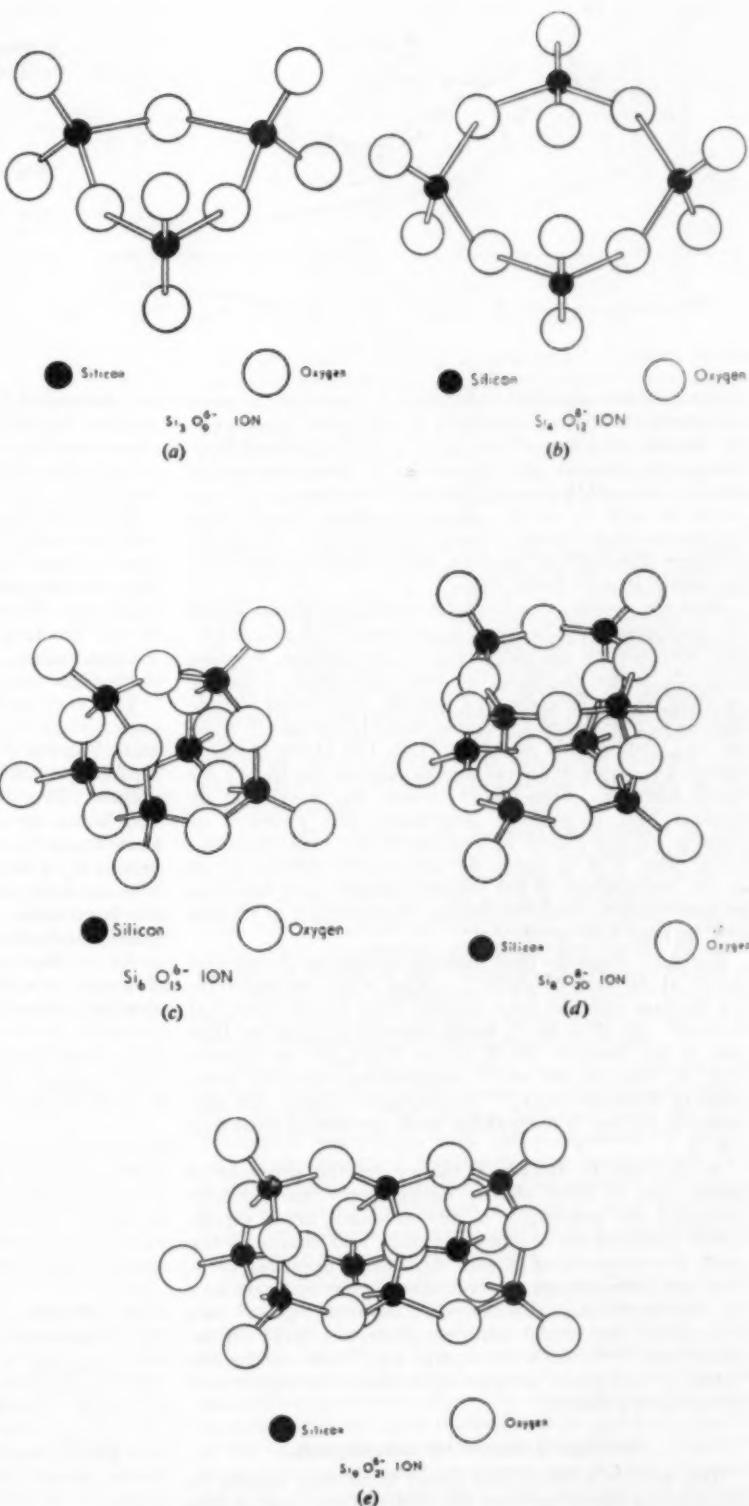


Fig. 3.—Some ions thought to exist in molten silicates.<sup>9b</sup>

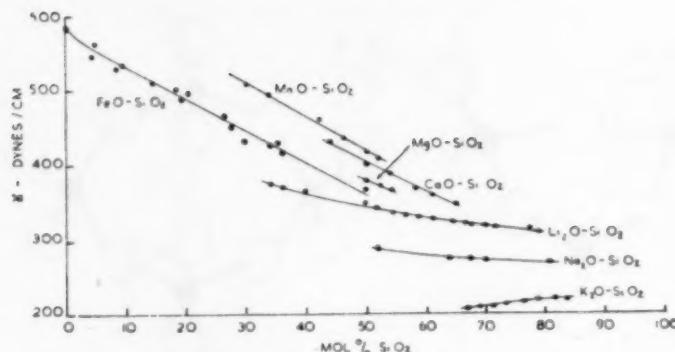


Fig. 4.—Surface tension versus composition for some binary silicates.<sup>15c</sup>

polymerisation occurred to form chains, until at the meta composition, chains of infinite length were postulated. On further reduction of the O : Si ratio, Endell and Hellbrügge postulated the formation of two-dimensional sheets, the completion of this structure being achieved at the 33 mole-% metal oxide composition. Any further decrease in metal oxide content resulted in the formation of three dimensional bonding, the structure eventually becoming that of fused silica.

Now the movement of infinite sheets and chains would be associated with a very large energy of activation. But the energy of activation *v* composition relation (Fig. 2) passes through the two compositions corresponding to the formation of infinite chains and infinite sheets, as postulated by Endell and Hellbrügge,<sup>10</sup> without any appreciable change. Thus, the above classical theory has now lost favour to the discrete ion theory for liquid silicates. This theory, which was developed by Bockris and co-workers, postulates the presence of discrete silicate anions at any particular concentration. These ions, it is thought, do not become infinite at all as the composition of the silicate changes, and hence at no composition could the energy of activation of viscous flow become very large.

Bockris<sup>11</sup> suggests that instead of infinite chains and sheets at 50 and 33 mole-% metal oxide respectively, the silicates exist as large anions, more or less spherical in shape. At 33 mole-% metal oxide it is suggested that ions of the formula  $(\text{Si}_6\text{O}_{15})^{6-}$  or  $(\text{Si}_9\text{O}_{20})^{8-}$  are present (Fig. 3), and at the meta composition, the ions form rings of formula  $(\text{Si}_4\text{O}_9)^6-$  or  $(\text{Si}_4\text{O}_{12})^{8-}$ . Thus, the discrete ion theory is compatible with the results shown in Fig. 2.

As opposed to Bockris<sup>11</sup> theory for the presence of single ions of fixed shape, Richardson<sup>12</sup> has recently indicated the possibility of the existence of an equilibrium distribution of silicate groups and chains in the melt, the proportions of each depending upon temperature and composition. As yet there seems to be no way of distinguishing conclusively between these two hypotheses, but recent infra-red absorption work on the phosphates,<sup>35,28</sup> which are similar long chain melts, has furnished a certain amount of evidence in support of Richardson's theory.

#### Surface Tension Measurements

The surface tension of a liquid is directly related to the binding forces between the constituents, and should therefore provide some structural information. In most cases, surface tension is by no means the best property to

be considered for a study of the constitution of substances, but the correct interpretation of surface tension measurements can reveal confirmatory evidence for a constitution which has been predicted from other considerations, e.g. viscosity and density measurements.

Boni and Derge<sup>13</sup> have reviewed the literature dealing with the surface tensions of molten silicates of metallurgical interest, and have indicated the useful generalisations which exist between surface tension, ionic potentials and types of chemical bonding. The type and strength of the bonding is very often reflected in the surface tension values. This is immediately evident on comparing the values given in Table I.

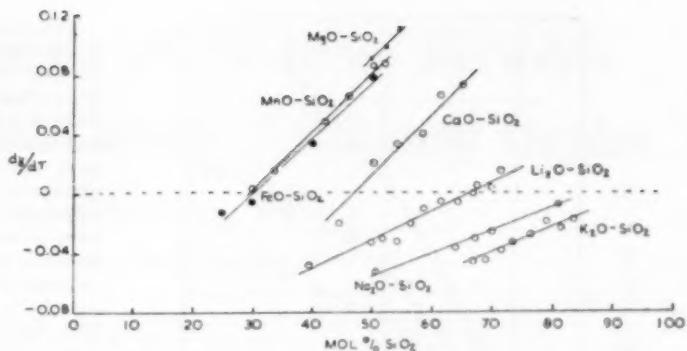
The very powerful metallic bond is reflected in the high surface tensions for the molten metals, whereas the weak Van der Waal forces known to exist in most organic liquids are reflected in their very low surface tension values. The surface tensions of the molecular liquids and liquid metals are respectively smaller and greater than those for the molten silicates, borates and phosphates by a factor of ten. The molten phosphates and silicates have values approaching the order of those for the fused salts. The general inference is that the phosphates and silicates are essentially ionic liquids.

As in the case of other physical properties, it is desirable to study and interpret the surface tensions of simpler melts before considering the more complex

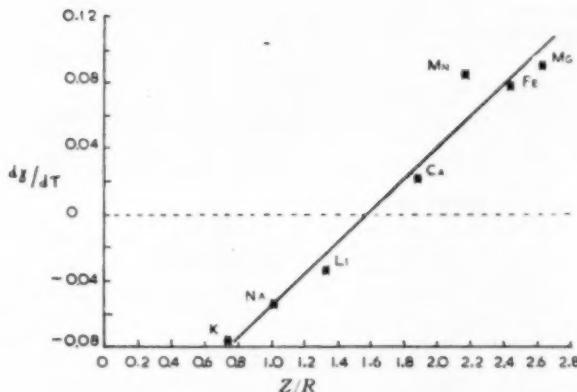
TABLE I

| Substance  | Surface Tension (dynes/cm.) | Temperature (°C.) |
|--|-----------------------------|-------------------|
| Si   | 1,615<br>(in helium)        | 1,470             |
| Fe   | 1,560<br>(in helium)        | 1,550             |
| Ca   | 600                         | 500               |
| FeO  | 584                         | 1,400             |
| Ca <sub>2</sub> S  | 410<br>(in argon)           | 1,130             |
| MnO <sub>2</sub> SiO <sub>3</sub>                            | 415                         | 1,570             |
| CaO <sub>2</sub> SiO <sub>3</sub>                            | 400                         | 1,570             |
| Na <sub>2</sub> O <sub>2</sub> SiO <sub>3</sub>              | 284                         | 1,400             |
| Na <sub>2</sub> O <sub>2</sub> P <sub>2</sub> O <sub>7</sub> | 177<br>(in nitrogen)        | 950               |
| CuCl <sub>2</sub>  | 145<br>(in argon)           | 800               |
| CuCl   | 92<br>(in argon)            | 450               |
| H <sub>2</sub> O   | 76                          | 0                 |
| CCl <sub>4</sub>   | 29                          | 0                 |

Fig. 5.— (i) (right)  $dy/dT$  versus composition for some binary silicates.<sup>15a</sup>



(ii) (below)  $dy/dT$  versus the ion-oxygen attraction parameter  $Z/e$ .<sup>15a</sup>



systems. Kozakevich<sup>14</sup> and King<sup>15a,b</sup> have measured the surface tensions of the binary silicates found in steel-making slags, e.g.  $FeO-SiO_2$  and  $MgO-SiO_2$ , and Shartsis and Spinner<sup>16</sup> have studied the alkali silicates. All these results have been obtained by a maximum pull-on-cylinder method. The findings of the above investigations have been summarised in a recent paper by King.<sup>15c</sup>

Fig. 4 which represents the work carried out by King<sup>15a</sup> and Shartsis and Spinner,<sup>16</sup> illustrates the effect of metal oxide additions on the surface tensions of the binary silicates. These relationships are all quite smooth, passing without inflection through the composition of the solid compounds. It is unsafe, however, to draw any conclusions from this observation alone concerning the existence of compounds in the liquid, since surface tension is not a very reliable property in this respect.

The linearity of the plots shown in Fig. 4 is compatible with the theory of a continuous and gradual variation in the structure of the melt. The most acceptable theory for the gradual increase in surface tension values on addition of metal oxide is that the degree of association is becoming less as the structure is progressively broken down. The silicate anions, as we have already seen, may be quite complex rings, chains, or even more complex anions, and, on addition of metal oxide, the number of smaller ionic entities will increase and this will result in an increased bond density near the surface, resulting in an increased surface tension. Since the driving force for this breakdown process is the ion-oxygen attraction of the cation, it is not surprising that there is a gradual increase in the surface tension values with a decrease in cation size, as indicated by the series of plots for different cations in Fig. 4.

King<sup>15a</sup> also observed a systematic increase in the temperature coefficient of surface tension values for the silicates as the ion-oxygen attraction for the cation increases. This effect is shown in Fig. 5, where it is evident that values tend to become abnormal, i.e. positive, as the ion-oxygen attraction increases. A similar tendency towards abnormality is shown as the proportion of silica increases.

Various mechanisms have been suggested to explain the existence of abnormal temperature coefficients of surface tension and these have been discussed in some detail by King<sup>15a</sup>. The most acceptable theory is that the abnormal  $dy/dT$  values are the result of a breakdown of the anion structure at higher temperatures into simpler units. The extent of this breakdown will be governed by the cation-oxygen attraction which supplies the driving force, and by the possible complexity of the structure, which depends on the silica content. The experimental results can be correlated quite well with this explanation.

In a recent study<sup>17</sup> of the surface tensions of some binary phosphates and silicates, it has been possible to develop a theoretical interpretation for the relationships shown in Fig. 5, and for similar relationships which have been obtained for the binary phosphates. Using a modified ion-oxygen attraction parameter, interpretations in terms of the variation of charge density on the silicate (and phosphate) networks have been developed, and this has enabled a comparison of the liquid silicate and phosphate structures to be made.

Barrett and Thomas<sup>18</sup> have recently measured the surface tensions of some melts in the ternary system  $CaO-Al_2O_3-SiO_2$ . The results obtained seem to be in reasonable agreement with those reported by King<sup>15a</sup> and Ermolaeva.<sup>19</sup> Surface-tension/temperature and surface-tension/composition relationships were all linear in form, and interpretations in terms of structure breakdown (or dissociation) were employed for the observed increases in surface tension values.

In conclusion, it seems that surface tension values for the silicates and the variation of these values with composition and temperature, can be correlated quite well with the theory of a dynamic equilibrium between the various possible silicate groupings. In general, the structure breakdowns effected by the addition of network modifiers to long chain melts result in marked increases in the surface tension values of these melts. Surface-tension measurements have shown that there is a relation between cation-oxygen attraction and surface tension and its temperature coefficient in binary silicates. From

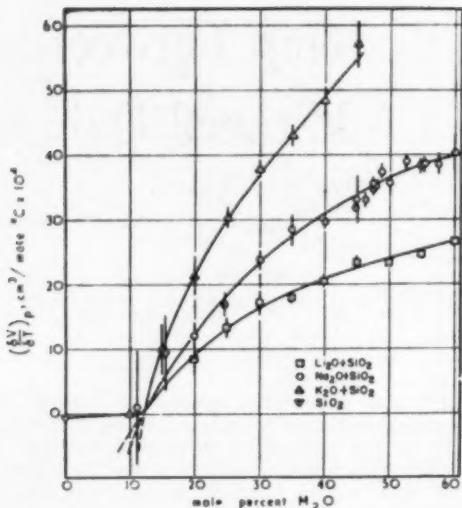


Fig. 6.—Thermal expansivities in simple binary silicates.<sup>21</sup>

this, and from the comparison between type of bonding and surface tension, confirmation of the ionic nature of silicate melts is indicated.

#### Density Measurements

Shartais, Spinner and Cappa<sup>20</sup> and Bockris, Tomlinson and White<sup>21</sup> have used a similar technique for measuring the densities of molten silicates. The method employed was a modification of the standard Archimedes principle for density measurements, using a platinum sinker. Due to the high viscosity values of the melts, these workers found it impossible to weigh the platinum sinker directly in the melt. The sinker was suspended in the melt by a fine stainless steel chain and platinum wire from one pan of an analytical balance. The sinker was set in motion in the melt by placing weights on the second pan, and its velocity was determined by observing, and timing the motion of the pointer across the graduated balance scale. The velocity of the sinker was varied by varying the load on the second balance pan, and a graph of weight on pan *v.* velocity of sinker was constructed. Extrapolation of this relationship to zero velocity gave a value for the weight of the sphere at rest in the melt. Using this value, the density of the melt could be calculated.

Bockris<sup>21</sup> and co-workers found that the interpretation of their results confirmed the general picture of the silicate structure as determined from viscosity measurements and which has been described previously. Shartais, Spinner and Cappa<sup>20</sup> did not attempt any detailed structural interpretation of their density results; they did, however, calculate the thermal expansivities of melts in the systems  $\text{Li}_2\text{O}-\text{SiO}_2$ ,  $\text{Na}_2\text{O}-\text{SiO}_2$  and  $\text{K}_2\text{O}-\text{SiO}_2$ , and observed that the expansivities of the potassium glasses were greater than those of the other two glasses. This effect can be accounted for by reference to the ion-oxygen attraction parameter concept. Since the ion-oxygen attraction is smaller for the  $\text{K}^+$  ion than for the  $\text{Na}^+$  and  $\text{Li}^+$  ions, the electrostatic forces operating between the metal ions and the silicate lattice are weaker in the potassium case. Hence, the lattice can expand more easily in the binary potassium silicate than in the sodium and lithium silicates.

The same binary silicates were studied by both sets of workers, but as a variation of experimental technique, Bockris<sup>21</sup> *et al* eliminated from their density calculation the surface tension correction for the pull on the suspension wire by the liquid surface. By measuring the loss in weight of two sinkers of different volumes in the melt, they obtained two simultaneous equations, and eliminated the value of the surface tension from these equations. Molar volumes and thermal expansivities were obtained from the density measurements. The thermal expansivities obtained for the three binary systems are shown in Fig. 6 and indicate a remarkable change in expansivity near 12 mole-% metal oxide. This change is commensurate with a structure breakdown in this region, since a loosely packed silicate structure will expand thermally, far more easily than a rigid and tightly bound structure.

(to be continued)

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#### Wellman-Schloemann Agreement

FOLLOWING negotiations finalised by Sir Peter Roberts, Bt., chairman of Wellman Smith Owen, on his recent visit to Düsseldorf, an agreement has been signed between Schloemann Aktiengesellschaft and The Wellman Smith Owen Engineering Corporation, Ltd., for collaboration in building rolling mills, hot and cold, ferrous and non-ferrous, for the United Kingdom and the Commonwealth. This will make available to British industry the modern technical advances in rolling mill design of Schloemann A.G. and the high class workmanship, experience and services of The Wellman Smith Owen Engineering Corporation, Ltd.

#### Winder Drive Order

THE GENERAL ELECTRIC CO., LTD., of England, has received a £50,000 order from the East Rand Engineering Co. of Johannesburg for equipment to be used in the conversion of a winder from steam to electric drive. The converted winder will be installed underground at the Witbank gold mine of the S.A. Rands and Exploration Co., Ltd., a subsidiary of the Anglo-American Corporation, Ltd. Twin 6.6 kV. 1,150 h.p. 10-pole A.C. motors will be used to drive the 12 ft. by 5 ft. double-drum winder which will be fitted with G.E.C.'s latest closed loop A.C. speed-control equipment. An unusual feature of the electrical system is that both rotors are to be connected in parallel to one liquid controller. Switch and control gear are included in the contract.

# An X-Ray Investigation of Systems Between Niobium Pentoxide and Certain Additional Oxides

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The binary constitutions of niobium pentoxide with the oxides of nickel, cobalt, iron, chromium, vanadium, titanium, zirconium, molybdenum, tungsten, magnesium, calcium, strontium, barium, cerium, aluminium and silicon have been studied on mixtures melted to varying compositions, and in a high and low temperature condition. The primary solid solubilities in  $\beta$ -Nb<sub>2</sub>O<sub>5</sub> are frequently extensive and accompanied by a degree of structural distortion. A number of intermediate niobates are formed for higher second-oxide contents and details have been determined; amongst these, the rutile, columbite, hematite, perovskite and other structure types enter dominantly, and their stability fields, transformations, lattice-dimensional and other changes are outlined. A major effect is that of the rutile and columbite-type niobates being in certain cases high and low temperature allotropic forms to one another. Several new compounds and new isomorphs to known structures were found. Homogeneity ranges, which sometimes are considerable and imply varying degrees of non-stoichiometry, are a characteristic of these phases, as well as of the primary end solid solutions. A systematic pattern of phase occurrences emerges and is discussed in the light of the periodic table and crystal chemistry.

(Continued from page 218 of the November issue).

## System Nb<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>

The constitutions observed in the system Nb<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub> are shown in Table VIII: the following conclusions may be drawn:—

- (a)  $\beta$ -Nb<sub>2</sub>O<sub>5</sub> dissolves large amounts of WO<sub>3</sub>, with a simultaneous small structural distortion. The solid solution extends to beyond 50% WO<sub>3</sub> at both high and low temperatures.
- (b) A new phase Y occurs at approximately Nb<sub>2</sub>O<sub>5</sub>-3WO<sub>3</sub>, which precipitates tungstic oxide at lower temperatures.
- (c) A limited solid solution of Nb<sub>2</sub>O<sub>5</sub> in WO<sub>3</sub> occurs, expanding the lattice. A check chemical analysis on the 75% WO<sub>3</sub> melt for a possible WO<sub>3</sub> loss during melting showed that this was unsubstantial.

Fig. 1h shows the phase relations diagrammatically.

## System Nb<sub>2</sub>O<sub>5</sub>-Ta<sub>2</sub>O<sub>5</sub>

The constitutions observed in the Ta<sub>2</sub>O<sub>5</sub>-Nb<sub>2</sub>O<sub>5</sub> system are shown in Table IX: the following conclusions may be drawn.

- (a) There exists an extensive solubility of Ta<sub>2</sub>O<sub>5</sub> in  $\beta$ -Nb<sub>2</sub>O<sub>5</sub>, somewhat above 10% Ta<sub>2</sub>O<sub>5</sub>; however,  $\alpha$ -Nb<sub>2</sub>O<sub>5</sub> then becomes stabilised. Tantalum pentoxide occurs in two allotropic modifications, the low temperature ( $\alpha$ ) form, isomorphous with  $\alpha$ -Nb<sub>2</sub>O<sub>5</sub>, and a high-temperature ( $\theta$ ) form, which is structurally distinct from  $\alpha$ . On adding Ta<sub>2</sub>O<sub>5</sub> to Nb<sub>2</sub>O<sub>5</sub>, an  $\alpha$ -(Ta,Nb)<sub>2</sub>O<sub>5</sub> solid solution appears at lower temperatures, in preference to  $\beta$ . This Ta-stabilised  $\alpha$ -Nb<sub>2</sub>O<sub>5</sub> transforms reversibly to the  $\theta$ -(Ta,Nb)<sub>2</sub>O<sub>5</sub> phase at high temperatures. The primary  $\theta$  and  $\alpha$ -Ta<sub>2</sub>O<sub>5</sub> solutions occur already for less than 50% Ta<sub>2</sub>O<sub>5</sub>.
- (b) No phases other than  $\beta$ -(Nb,Ta)<sub>2</sub>O<sub>5</sub>,  $\alpha$ -(Ta,Nb)<sub>2</sub>O<sub>5</sub> and  $\theta$ -(Ta,Nb)<sub>2</sub>O<sub>5</sub> were observed in equilibrium in this system, and it should be noted that, whilst for pure Nb<sub>2</sub>O<sub>5</sub> the  $\alpha$  form has been proved to transform irreversibly,<sup>1</sup> this transformation becomes rever-

TABLE VIII.—SYSTEM Nb<sub>2</sub>O<sub>5</sub>-WO<sub>3</sub>

| Composition<br>(Molecular<br>% WO <sub>3</sub> ) | Heat<br>Treatment | Constitution  | Remarks   |
|--|-------------------|---|---|
| 10%  | As melted         | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                |   |
| 10%  | 800° C., 14 days  | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                |   |
| 25%  | As melted         | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                |   |
| 25%  | 800° C., 14 days  | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                |   |
| 50%  | As melted         | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                | Small differences in fine structure, and small contraction of lattice on annealing. Continuous minor variations in line grouping and relative intensities occur within solid solution |
| 50%  | 800° C., 14 days  | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                |   |
| 62.5%  | As melted         | $\beta$ -Nb <sub>2</sub> O <sub>5</sub> solid solution, pure                | No Y yet  |
| 62.5%  | 800° C., 14 days  | Y-phase, very large<br>$\beta$ -Nb <sub>2</sub> O <sub>5</sub> , very small | Y = new phase   |
| 70%  | As melted         | Y, pure   |   |
| 70%  | 800° C., 14 days  | Y, pure   |   |
| 75%  | As melted         | Y, large<br>WO <sub>3</sub> , small   |   |
| 75%  | 800° C., 14 days  | Y, large/medium<br>WO <sub>3</sub> , medium                                 |   |
| 90%  | As melted         | WO <sub>3</sub> , large<br>Y, small   |   |
| 90%  | 800° C., 14 days  | WO <sub>3</sub> , solid solution, pure                                      | Lattice expanded through Nb <sub>2</sub> O <sub>5</sub>   |
| 100%   | As melted         | WO <sub>3</sub> (perovskite type)   |   |
| 100%   | 800° C., 14 days  | WO <sub>3</sub>   | No change   |

sible on tantalum addition; this applies both to the  $\alpha \rightarrow \beta$  change at the Nb-end, and the  $\alpha \rightarrow \theta$  change at the Ta-end. The existence of the high-temperature ( $\theta$ ) form of Ta<sub>2</sub>O<sub>5</sub> had already been reported by Lagergren and Magneli<sup>7</sup>, but was later denied by Schönberg.<sup>8</sup> It is here clearly confirmed. The  $\theta$ -form is considered of particular interest in

TABLE IX.—SYSTEM  $\text{Nb}_2\text{O}_5$ — $\text{Ta}_2\text{O}_5$ 

| Composition (Molecular % $\text{Ta}_2\text{O}_5$ ) | Heat Treatment   | Constitution  | Remarks   |
|--|------------------|---|---|
| 10%  | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ solid solution, pure  |   |
| 10%  | 800° C., 14 days | Same  |   |
| 20%  | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ , medium<br>$\theta$ ( $\text{Ta}_2\text{Nb}_2\text{O}_5$ ), medium/large   | $\theta$ = high temperature form of $\text{Ta}_2\text{O}_5$ |
| 20%  | 800° C., 14 days | $\alpha$ - $(\text{Ta}_2\text{Nb}_2\text{O}_5)$ , medium<br>$\theta$ ( $\text{Ta}_2\text{Nb}_2\text{O}_5$ ), medium<br>$\beta$ - $\text{Nb}_2\text{O}_5$ solid solution, medium/small |   |
| 25%  | As melted        | $\theta$ - $(\text{Ta}_2\text{Nb}_2\text{O}_5)$ , very large<br>$\beta$ - $\text{Nb}_2\text{O}_5$ , small   |   |
| 25%  | 800° C., 14 days | $\alpha$ - $\text{Nb}_2\text{O}_5$ , large<br>$\beta$ - $\text{Nb}_2\text{O}_5$ , medium  |   |
| 50%  | As melted        | $\theta$ - $(\text{Ta}_2\text{Nb}_2\text{O}_5)$ , pure  |   |
| 50%  | 800° C., 14 days | $\alpha$ - $(\text{Ta}_2\text{Nb}_2\text{O}_5)$ , pure  |   |
| 100%   | As melted        | $\theta$ - $\text{Ta}_2\text{O}_5$ , pure   |   |
| 100%   | 800° C., 14 days | $\alpha$ - $\text{Ta}_2\text{O}_5$ , pure   |   |

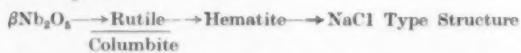
relation to Nb-base alloys, because it could be retained to lower temperatures at higher Nb-contents.

A schematic diagram is shown in Fig. 1*i*.

### System $\text{Nb}_2\text{O}_5$ — $\text{MgO}$

The constitutions observed in the  $\text{Nb}_2\text{O}_5$ — $\text{MgO}$  system are shown in Table X: the following conclusions may be drawn:—

- (a)  $\text{MgO}$  forms an appreciable solid solution in  $\beta$ - $\text{Nb}_2\text{O}_5$ , somewhat below 10%;  $\alpha$ - $\text{Nb}_2\text{O}_5$  is not stabilised.
- (b) The rutile structure (at high temperatures) and the columbite structure (at low temperatures) follow  $\beta$ - $\text{Nb}_2\text{O}_5$ , columbite possessing a considerable homogeneity field. In contrast to the  $\text{NiO}$ ,  $\text{CoO}$ , and  $\text{FeO}$ -base columbite, stable only at low temperatures, the  $\text{MgO}$  columbite appears already at higher temperatures, coexisting with rutile. Furthermore a partial decomposition  $\text{R} \rightarrow \text{C} + \beta$ - $\text{Nb}_2\text{O}_5$  takes place, indicating marked composition differences between  $\text{R}$  and  $\text{C}$ . An  $\text{MgO}$ - $\text{Nb}_2\text{O}_5$  rutile has not previously been reported (in contrast to those with  $\text{NiO}$ ,  $\text{CoO}$  and  $\text{FeO}$ ); a columbite  $\text{MgO}$ - $\text{Nb}_2\text{O}_5$  is known, however (Brandt),<sup>12</sup> though the composition here diverges.
- (c) The hematite structure occurs for  $\text{MgO}$ -richer compositions, followed by cubic  $\text{MgO}$  proper. Thus the sequence



appears consistently once again in this system.

The hematite phase has a finite homogeneity range; it is capable of precipitating free  $\text{MgO}$  at lower temperatures, under change of unit cell dimensions. Cubic  $\text{MgO}$  can take a small amount of  $\text{Nb}_2\text{O}_5$  into solution, contracting in doing so (an unusual behaviour considering atomic sizes, but probably reflecting increase in metal atom vacancies, similarly to  $\text{CoO}$ ).

The schematic overall diagram is shown in Fig. 1*j*.

### System $\text{Nb}_2\text{O}_5$ — $\text{CaO}$

The constitutions observed in the  $\text{Nb}_2\text{O}_5$ — $\text{CaO}$  system

are shown in Table XI: the following conclusions may be drawn:—

- (a) A limited solid solution occurs of  $\text{CaO}$  in  $\beta$ - $\text{Nb}_2\text{O}_5$ , with a small amount of lattice-distortion.
- (b) Three calcium niobates occur, structurally distinct: a compound  $\text{I}_{\text{Ca}}$  at about  $\text{CaO} \cdot \text{Nb}_2\text{O}_5$  (nominal); a compound  $\text{Q}$  at about  $3\text{CaO} \cdot \text{Nb}_2\text{O}_5$ ; and a compound  $\text{P}$  of perovskite type of structure, closely adjoining  $\text{Q}$ , at about  $4\text{CaO} \cdot \text{Nb}_2\text{O}_5$ . Marked subsidiary distortions of  $\text{P}$  and of  $\text{Q}$  occur with composition and heat treatment. Phase  $\text{Q}$  is a "niobium bronze" related to the alkali tungsten bronzes (sodium tungstates of variable composition  $\text{Na}_x\text{WO}_3$  ( $X < 1$ )); structurally this conforms with its continuity with perovskite, because the defect lattices involved during sodium subtraction and characteristic of this family terminate in the perovskite configuration; the perovskite type structure is apparent already from the diffraction pattern of  $\text{Q}$ , but the spacing is considerably decreased for lesser  $\text{CaO}$  (again consistent with the subtractive lattice). The present perovskite equilibria are complex and require further study (as arises e.g. from the co-existence with some free  $\text{CaO}$  whilst the perovskite dimensions change) and a concealed peritectic reaction seems possible. The perovskite lattice expands with  $\text{CaO}$  content, and the degree of tetragonality varies.

TABLE X.—SYSTEM  $\text{Nb}_2\text{O}_5$ — $\text{MgO}$ 

| Composition (Molecular % $\text{MgO}$ ) | Heat Treatment               | Constitution  | Remarks  |
|---|------------------------------|---|--|
| 10%                                     | As melted                    | $\beta$ - $\text{Nb}_2\text{O}_5$ , very large<br>$\text{R}$ (= rutile), trace    |  |
| 10%                                     | 800° C., 14 days             | $\beta$ - $\text{Nb}_2\text{O}_5$ , very large<br>$\text{C}$ (= columbite), trace |  |
| 25%                                     | As melted                    | $\beta$ - $\text{Nb}_2\text{O}_5$ , large/medium<br>$\text{R}$ , medium           |  |
| 25%                                     | 800° C., 14 days             | $\beta$ - $\text{Nb}_2\text{O}_5$ , large<br>$\text{C}$ , medium/small            |  |
| 50%                                     | As melted                    | $\text{R}$ , large<br>$\text{C}$ , medium   | $\text{R}$ -spacings:<br>$a = 4 \cdot 739 \text{ kX}$<br>$c = 3 \cdot 063 \text{ kX}$                                  |
| 50%                                     | 800° C., 14 days             | $\text{C}$ , large<br>$\beta$ - $\text{Nb}_2\text{O}_5$ , small                   | $\text{C}$ -spacings:<br>$a = 5 \cdot 162 \text{ kX}$<br>$b = 4 \cdot 174 \text{ kX}$<br>$c = 5 \cdot 339 \text{ kX}$  |
| 75%                                     | As melted                    | $\text{R}$ , large/medium<br>$\text{C}$ , large/medium                            | $\text{R}$ -spacings:<br>$a = 4 \cdot 743 \text{ kX}$<br>$c = 3 \cdot 059 \text{ kX}$                                  |
| 75%                                     | 800° C., 14 days             | $\text{C}$ , large<br>$\beta$ - $\text{Nb}_2\text{O}_5$ , small                   | $\text{C}$ -spacings:<br>$a = 5 \cdot 015 \text{ kX}$<br>$b = 14 \cdot 184 \text{ kX}$<br>$c = 5 \cdot 861 \text{ kX}$ |
| 90%                                     | As melted                    | $\text{H}$ (= hematite), large<br>$\text{MgO}$ , small                            |  |
| 90%                                     | 800° C., 14 days             | $\text{H}$ , large<br>$\text{MgO}$ , small  | $\text{H}$ -spacing decreases on annealing   |
| 95%                                     | As melted                    | $\text{H}$ , large<br>$\text{MgO}$ , medium                                       | $\text{H}$ -spacing changes from:<br>$a = 5 \cdot 160 \text{ kX}$<br>$c = 14 \cdot 003 \text{ kX}$                     |
| 95%                                     | 800° C., 14 days             | $\text{H}$ , large/medium<br>$\text{MgO}$ , medium                                | to:<br>$a = 5 \cdot 147 \text{ kX}$<br>$c = 14 \cdot 090 \text{ kX}$<br>on annealing                                   |
| 98%                                     | Sample sintered at 1,700° C. | $\text{MgO}$ , large<br>$\text{H}$ , medium                                       | $a = 4 \cdot 180 \text{ kX}$   |
| 98%                                     | 800° C., 14 days             | Same  |  |
| 100%                                    | —                            | $\text{MgO}$  | $a = 4 \cdot 205 \text{ kX}$   |

(c) No solid solution of  $\text{Nb}_2\text{O}_5$  in  $\text{CaO}$  occurs.

Note.—A lime-niobate  $\text{CaNbO}_3$  (and other "niobium bronzes" analogous to tungsten bronzes) has been reported by Krylov:<sup>9</sup> a subtractive lattice can here be formed, and as a representative perovskite composition  $\text{Ca}_{0.82}\text{NbO}_3$  was given; the present compound of this structure seems rather higher in  $\text{Ca}$ , but could represent a replacement series of solid solutions with Krylov's phase. Stoichiometrically the composition  $4\text{CaO} \cdot \text{Nb}_2\text{O}_5$  ( $= \text{Me}_2\text{O}_3$ ) would be quite consistent, but a lower  $\text{Ca}/\text{Nb}$  ratio is possible (a reservation which must apply throughout this system).

The schematic overall diagram is shown in Fig. 1k.

The pyrochlore family of mineral structures might well have entered in the present system; general composition:

( $\text{Na, K, Ca, Fe, Ce, Y, Th, U et al.}_3$  ( $\text{Nb, Ta, Ti, Fe et al.}_2$  ( $\text{O, OH, F}_7$ , i.e.  $\text{A}_3 \text{B}_2 \text{X}_7$ )

This has of course been much studied; the structure is cubic, and the spacing variable (but of order  $a = 10.3\text{kX}$ ). Gärtner<sup>10</sup> reported pyrochlore and perovskite to form partial solutions, but, upon check, the present three  $\text{CaO} \cdot \text{Nb}_2\text{O}_5$  phases do not agree with pyrochlore, and other radicles are evidently required for its stabilisation.

TABLE XI.—SYSTEM  $\text{Nb}_2\text{O}_5\text{-CaO}$

| Composition (Molecular % $\text{CaO}$ ) | Heat Treatment   | Constitution  | Remarks  | 35-6% | 800° C., 14 days | Same                       |                                  |
|---|------------------|---|--|-------|------------------|----------------------------|----------------------------------|
| 10%                                     | As melted        | $\beta$ : $\text{Nb}_2\text{O}_5$ , large<br>New phase $\text{I}_{\text{Ca}}$ , small |  | 51%   | As melted        | P (= perovskite), pure     | Spacing:<br>$a = 4.123\text{kX}$ |
| 10%                                     | 800° C., 14 days | $\beta$ , large<br>$\text{I}_{\text{Ca}}$ , small                                     | Small distortion in $\beta$ -structure   | 51%   | 800° C., 14 days | Same                       |                                  |
| 25%                                     | As melted        | $\beta$ , large<br>$\text{I}_{\text{Ca}}$ , small/medium                              | Main difference from 10%: $\text{CaO}$ was slight further change in line-separation, i.e. further solution in $\beta$  | 69%   | As melted        | Large $\text{SrO}$ , small | $a = 4.145\text{kX}$             |
| 25%                                     | 800° C., 14 days | $\beta$ , large<br>$\text{I}_{\text{Ca}}$ , small/medium                              |  | 69%   | 800° C., 14 days | Same                       |                                  |
| 50%                                     | As melted        | $\text{I}_{\text{Ca}}$ , pure   | $\text{I}_{\text{Ca}} = \text{CaO} \cdot \text{Nb}_2\text{O}_5$ ( $= \text{CaNb}_2\text{O}_5$ )  |       |                  |                            |                                  |
| 50%                                     | 800° C., 14 days | $\text{I}_{\text{Ca}}$ , pure   | No change  |       |                  |                            |                                  |
| 62.5%                                   | As melted        | $\text{I}_{\text{Ca}}$ , large/medium   |  |       |                  |                            |                                  |
| 62.5%                                   | 800° C., 14 days | —   |  |       |                  |                            |                                  |
| 75%                                     | As melted        | New phase Q   | Trace perovskite possible  |       |                  |                            |                                  |
| 75%                                     | 800° C., 14 days | Q   | No change  |       |                  |                            |                                  |
| 77.5%                                   | As melted        | Q (slightly varied intensities)   | $\text{Q}$ is already perovskite derivative (tetragonal bronze type). Continuous change in axial ratio of perovskite with $\text{Nb}/\text{Ca}$ ratio, from 75 to 90% $\text{CaO}$ |       |                  |                            |                                  |
| 77.5%                                   | 800° C., 14 days | Same  |  |       |                  |                            |                                  |
| 80%                                     | As melted        | P (= perovskite), large $\text{CaO}$ , small  | $a = 3.985\text{kX}$<br>$c = 3.946\text{kX}$   |       |                  |                            |                                  |
| 80%                                     | 800° C., 14 days | Same  | $a = 3.983\text{kX}$<br>$c = 3.948\text{kX}$   |       |                  |                            |                                  |
| 85%                                     | As melted        | P, large $\text{CaO}$ , medium  | $a = 3.992\text{kX}$<br>$c = 3.912\text{kX}$   |       |                  |                            |                                  |
| 85%                                     | 800° C., 14 days | Same  | $a = 3.996\text{kX}$<br>$c = 3.920\text{kX}$   |       |                  |                            |                                  |
| 90%                                     | As melted        | P, large/medium   | $a = 4.000\text{kX}$<br>$c = 3.986\text{kX}$   |       |                  |                            |                                  |
| 90%                                     | 800° C., 14 days | $\text{CaO}$ , medium<br>Same   | $a = 3.99\text{kX}$<br>$c = 3.923\text{kX}$  |       |                  |                            |                                  |
| 100%                                    | As melted        | $\text{CaO}$  | $\text{CaO}$ spacing unaltered through $\text{Nb}_2\text{O}_5$   |       |                  |                            |                                  |

TABLE XII.—SYSTEM  $\text{Na}_2\text{O}_5\text{-SrO}$

| Composition (Molecular % $\text{SrO}$ ) | Heat Treatment   | Constitution  | Remarks   |
|---|------------------|---|---|
| 1-3%                                    | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ , large<br>$\text{I}_{\text{Ba}}$ , small         | $\text{I}_{\text{Ba}}$ = new intermediate phase isomorphous with one observed in barium niobate |
| 1-3%                                    | 800° C., 14 days | $\beta$ - $\text{Nb}_2\text{O}_5$ , large<br>$\text{I}_{\text{Ba}}$ , small/medium  |   |
| 3-8%                                    | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ , large/medium<br>$\text{I}_{\text{Ba}}$ , medium | $\beta$ lattice slightly modified through solution  |
| 3-8%                                    | 800° C., 14 days | $\beta$ - $\text{Nb}_2\text{O}_5$ , medium<br>$\text{I}_{\text{Ba}}$ , medium       |   |
| 7-3%                                    | As melted        | $\text{I}_{\text{Ba}}$ , almost pure  |   |
| 7-3%                                    | 800° C., 14 days | Same  |   |
| 10-6%                                   | As melted        | $\text{I}_{\text{Ba}}$ , medium<br>$\text{J}_{\text{Ba}}$ , medium/small            | $\text{J}_{\text{Ba}}$ = further new phase possessing a Ba-isomorph                             |
| 10-6%                                   | 800° C., 14 days | Same  |   |
| 28%                                     | As melted        | $\text{J}_{\text{Ba}}$ , pure   |   |
| 28%                                     | 800° C., 14 days | Same  |   |
| 35-6%                                   | As melted        | $\text{K}_{\text{Ba}}$ , large<br>$\text{J}_{\text{Ba}}$ , small                    | $\text{K}_{\text{Ba}}$ = further new phase possessing a Ba-isomorph                             |
| 35-6%                                   | 800° C., 14 days | Same  |   |

For higher  $\text{SrO}$  contents hydration difficulties arose.

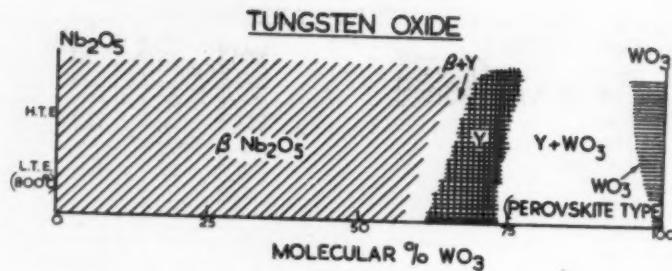
Note.—Some difficulty was experienced in this system through the raw strontium oxide being unavoidable in the hydrated form and an appropriate conversion in composition had to be applied. The precise composition figures quoted should not be stressed, but rather the constitutional sequence in principle; this holds also for the  $\text{CaO}$  and  $\text{BaO}$  systems.

### System $\text{Nb}_2\text{O}_5\text{-SrO}$

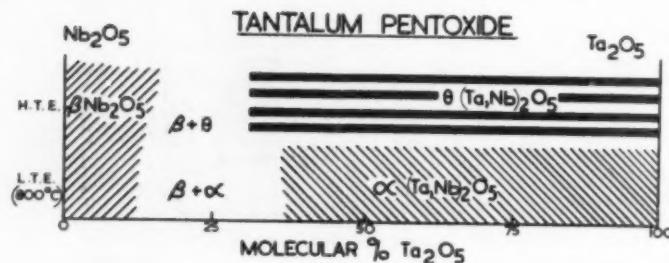
The constitutions observed in the system  $\text{Nb}_2\text{O}_5\text{-SrO}$  are shown in Table XII: the following conclusions may be drawn:

- The solid solubility of  $\text{SrO}$  in  $\beta\text{-Nb}_2\text{O}_5$  is small but significant. It decreases at lower temperatures.
- Three strontium niobates occur between  $\text{Nb}_2\text{O}_5$  and perovskite, denoted  $\text{I}_{\text{Ba}}$ ,  $\text{J}_{\text{Ba}}$  and  $\text{K}_{\text{Ba}}$ , being isomorphous with phases in the  $\text{Nb}_2\text{O}_5\text{-BaO}$  system, but not with those in  $\text{Nb}_2\text{O}_5\text{-CaO}$ . The fourth phase, P, of perovskite-type structure occurs at  $\text{SrO} \cdot \text{Nb}_2\text{O}_5$ , and is of course an analogue to both the  $\text{CaO}$  and  $\text{BaO}$  perovskites. This perovskite is cubic, as against the  $\text{CaO}$  one, and a finite homogeneity range is reflected in an increase in lattice-spacing with  $\text{SrO}$  content. The approximate compositions (scaled to 3 oxygen atoms as in the perovskite unit cell) are for  $\text{I}_{\text{Ba}}$ :  $\text{Nb}_{1.2}\text{Sr}_{0.05}\text{O}_3$ ; for  $\text{J}_{\text{Ba}}$ :  $\text{Nb}_{1.1}\text{Sr}_{0.2}\text{O}_3$ ; for  $\text{K}_{\text{Ba}}$ :  $\text{Nb}_{1.1}\text{Sr}_{0.35}\text{O}_3$ ; for perovskite:  $\text{Nb}_{0.5}\text{Sr}_{0.5}\text{O}_3$ . There is a remarkable independence in constitution with heat treatment throughout this system.
- $\text{SrO}$  and perovskite are in direct equilibrium (as for  $\text{CaO}$  and  $\text{BaO}$ ).
- No solid solution occurs of  $\text{Nb}_2\text{O}_5$  in  $\text{SrO}$ .

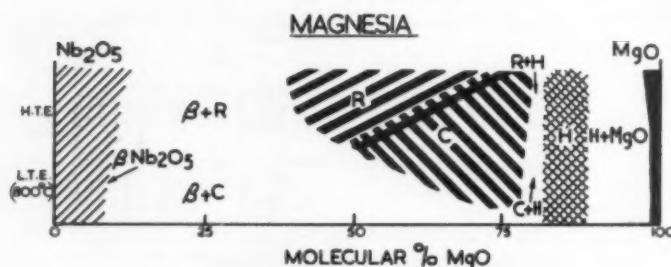
Fig. 11 shows the relevant diagram. (See also note at end of next section).



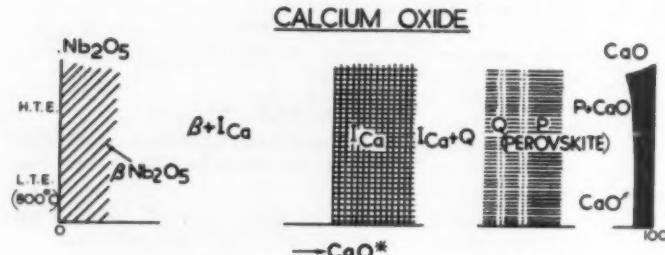
(h) System  $\text{Nb}_2\text{O}_5\text{-WO}_3$



(i) System  $\text{Nb}_2\text{O}_5\text{-Ta}_2\text{O}_5$



(j) System  $\text{Nb}_2\text{O}_5\text{-MgO}$ .



(k) System  $\text{Nb}_2\text{O}_5\text{-CaO}$ .

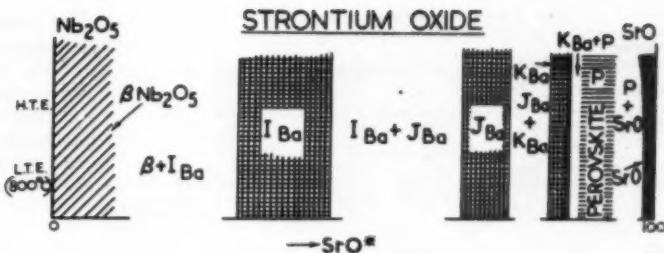
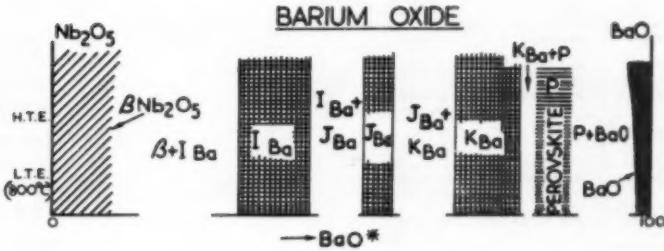
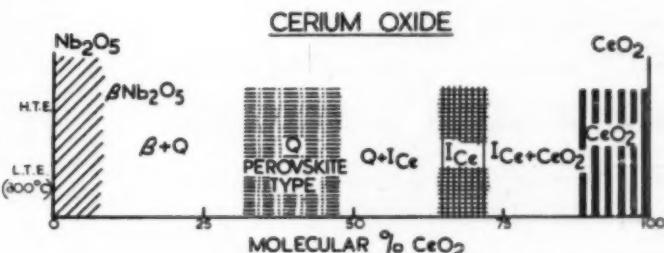
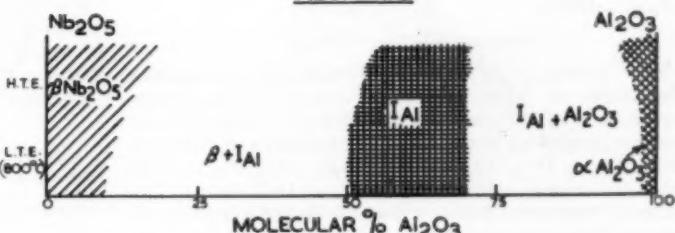
KEY TO STRUCTURE TYPES AND SYMBOLS ( )

|                                |   |   |
|--------------------------------|---|---|
| $\beta \text{Nb}_2\text{O}_5$  | = | (β)   |
| $\alpha \text{Nb}_2\text{O}_5$ | = | (α)   |
| RUTILE                         | = | (R)   |
| NIOBITE (COLUMBITE)            | = | (C)   |
| HEMATITE                       | = | (H)   |
| SODIUM CHLORIDE                | = | ■   |
| SPINEL                         | = |   |
| $\beta$ DERIVATIVES            | = | (β' FOR $\beta'(\text{V}, \text{Nb})_2\text{O}_5$ ;<br>D FOR $\text{Nb}_2\text{TiO}_7$ = DYSON PHASE) |
| PEROVSKITE & DERIVATIVES       | = | TUNGSTEN BRONZE<br>(P) CLASS (Q)  |

\* = COMPOSITIONS LEFT OPEN

- =  $\theta \text{ Ta}_2\text{O}_5$  (θ)
- = CUBIC ZIRCONIA ( $z_C$ )
- = MONOCLINIC ZIRCONIA ( $z_M$ )
- = FLUORSPAR
- = ANY OTHER STRUCTURE  
(y: (Nb, W) OXIDE)
- $I_X$  = INTERMEDIATE OXIDES  
FOR X = Ca, Sr, Ba, Al and Ce)

Fig. 1. Present constitutional results of systems of niobium pentoxide with second oxides.

(l) System  $\text{Nb}_2\text{O}_5$ -SrO.(m) System  $\text{Nb}_2\text{O}_5$ -BaO.(n) System  $\text{Nb}_2\text{O}_5$ -CeO<sub>2</sub>.(o) System  $\text{Nb}_2\text{O}_5$ -Al<sub>2</sub>O<sub>3</sub>.

### System $\text{Nb}_2\text{O}_5$ -BaO

Work on this system is still proceeding, and some uncertainty in absolute compositions exists at present. However, the following conclusions can be drawn at this stage :—

- $\beta$ - $\text{Nb}_2\text{O}_5$  possesses a significant solubility for barium oxide, which decreases with temperature, and involves a small lattice distortion, as for the other alkaline earths.
- There are three barium niobates, denoted (with increasing BaO content):  $I_{\text{Ba}}$ ,  $J_{\text{Ba}}$  and  $K_{\text{Ba}}$ . They are isomorphous to the strontium niobates referred to above, and of appreciably larger lattice-dimensions.
- These are followed by a perovskite-type phase, as for SrO and CaO, which itself coexists with barium oxide.

In composition these phases are probably similar to the strontium niobates, but with reservations; defect lattice formation is certainly a major feature. The phase relations are shown schematically in Fig. 1m.

The "niobium bronzes" also are known to include strontium and barium niobates as well as calcium and alkali ones. A fuller list, according to Krylov<sup>9</sup> is:  $\text{LiNbO}_3$ ,  $\text{Na}_4\text{Nb}_3\text{O}_8$ ,  $\text{K}_2\text{Nb}_2\text{O}_5$ ,  $\text{CaNbO}_3$ ,  $\text{SrNbO}_3$  and  $\text{BaNbO}_3$ . These, it will be noted, involve  $\text{NbO}_2$  rather than  $\text{Nb}_2\text{O}_5$ , and are liable to be promoted under reducing conditions. Exact chemical formulae for these bronzes, as for tungsten bronzes and the basic perovskite, cannot be established, owing to the variation of number of atoms per unit cell.

A strontium niobium bronze of composition range  $\text{Sr}_{0.7}\text{NbO}_3$ - $\text{Sr}_{0.9}\text{NbO}_3$  has also been reported by Ridgley and Ward.<sup>11</sup> These are cubic perovskites, but

TABLE XIII.—SYSTEM  $\text{Nb}_2\text{O}_5$ —(Ce, R.E.) $\text{O}_3$ 

| Composition (Molecular % (Ce, R.E.) $\text{O}_3$ ) | Heat Treatment   | Constitution   | Remarks   |
|--|------------------|--|---|
| 10%  | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ , large<br>Phase $Q$ , small       |   |
| 25%  | As melted        | $Q$ , large<br>$\beta$ , small/medium                                | $Q$ of perovskite type structure (though bronze derivative) |
| 25%  | 800° C., 14 days | $Q$ , large<br>$\beta$ , small/medium (slightly less than as melted) |   |
| 40%  | As melted        | $Q$ , pure   | $a = 3.233\text{kX}$  |
| 40%  | 800° C., 14 days | $Q$ , pure   | $c = 5.265\text{kX}$  |
| 50%  | As melted        | $Q$ , large<br>$I_{Ce}$ , small/medium                               | $I_{Ce}$ = new phase  |
| 50%  | 800° C., 14 days | $Q$ , large<br>$I_{Ce}$ , small/medium                               | —   |
| 62.5%  | As melted        | $Q$ , large<br>$I_{Ce}$ , very small                                 |   |
| 62.5%  | 800° C., 14 days | $Q$ , large<br>$I_{Ce}$ , very small                                 |   |
| 75%  | As melted        | $I_{Ce}$ , almost pure<br>$\text{CeO}_3$ , trace                     |   |
| 75%  | 800° C., 14 days | $I_{Ce}$ , large<br>$\text{CeO}_3$ , small/medium                    |   |
| 82.5%  | As melted        | $\text{CeO}_3$ solid solution, large<br>$I_{Ce}$ , medium            | $a = 5.458\text{kX}$  |
| 82.5%  | 800° C., 14 days | $\text{CeO}_3$ solid solution, large<br>$I_{Ce}$ , medium            | $a = 5.429\text{kX}$  |
| 90%  | As melted        | $\text{CeO}_3$ solid solution, pure                                  | $a = 5.458\text{kX}$  |
| 90%  | 800° C., 14 days | $\text{CeO}_3$ solid solution, pure                                  | $a = 5.418\text{kX}$  |
| 100%   | —                | $\text{CeO}_3$   | $a = 5.396\text{kX}$  |

degenerate into distorted lattices for Sr-deficiency ( $\text{Sr}_{0.45-0.5}\text{NbO}_3$ ).

#### System $\text{Nb}_2\text{O}_5$ —“ $\text{CeO}_2$ ”

The “cerium oxide” employed contained considerable admixtures of other rare earth oxides and is better described as of  $\text{CeO}_2$ -base or (Ce, R.E.) $\text{O}_3$ ; approximate analysis, according to the suppliers, Hopkin and Williams Ltd.:  $\text{La}_2\text{O}_3$ —16%,  $\text{Pr}_2\text{O}_3$ —19%,  $\text{Nd}_2\text{O}_3$ —13%,  $\text{Sm}_2\text{O}_3$ —2%; other rare earth oxides—4% (balance)  $\text{CeO}_2$ —46%. The material had nevertheless the single fluorite type structure of  $\text{CeO}_2$ , and the effect of such a typical technical rare earth oxide upon  $\text{Nb}_2\text{O}_5$  was of interest; “ $\text{CeO}_2$ ” below should be read with this understanding.

The constitutions of the system  $\text{Nb}_2\text{O}_5$ — $\text{CeO}_2$  are shown in Table XIII: the following conclusions may be drawn:—

- A restricted solid solution occurs of  $\text{CeO}_2$  in  $\beta$ - $\text{Nb}_2\text{O}_5$ , estimated as in the order of 5%  $\text{CeO}_2$ .
- Two compounds occur, one ( $Q$ ) of the tetragonal bronze class associated with perovskite, the other ( $I_{Ce}$ ) of a new structure. The approximate compositions are:

$$Q = 3\text{Nb}_2\text{O}_5 \cdot 2\text{CeO}_2$$

$$I_{Ce} = 3\text{Nb}_2\text{O}_5 \cdot 7\text{CeO}_2$$

- A considerable solid solubility exists of  $\text{Nb}_2\text{O}_5$  in  $\text{CeO}_2$ —up to about 12%  $\text{Nb}_2\text{O}_5$ . The lattice of  $\text{CeO}_2$  (cubic  $\text{CaF}_2$  type structure) is expanded through  $\text{Nb}_2\text{O}_5$  addition; the solid solution is contracted on annealing.

The manner of ionic replacement by which such expansion of  $\text{CeO}_2$  by  $\text{Nb}_2\text{O}_5$  could occur is of some considerable interest, because non-stoichiometry in the fluorite structure of  $\text{CeO}_2$  is known (e.g. on  $\text{CeO}_2 \rightarrow \text{La}_2\text{O}_3$  replacement), and it seems probable that  $\text{Nb}_2\text{O}_5$  solution involves to a large measure occupation of vacant lattice sites, apart from substitutions. The diagram is shown in Fig. 1n.

#### System $\text{Nb}_2\text{O}_5$ — $\text{Al}_2\text{O}_3$

The constitutions found on this system are shown in Table XIV: the following conclusions may be drawn:—

- $\text{Al}_2\text{O}_3$  enters into an extensive solid solution in  $\beta$ - $\text{Nb}_2\text{O}_5$  up to the order of 15%  $\text{Al}_2\text{O}_3$  in melting equilibrium. The  $\beta$  phase-boundary recedes with decreasing temperature.
- One compound was observed:  $I_{Al}$  of approximate composition  $\text{Al}_2\text{O}_3 \cdot \text{Nb}_2\text{O}_5$  ( $\text{AlNbO}_4$ ). This is in equilibrium with the primary  $\text{Nb}_2\text{O}_5$  and  $\text{Al}_2\text{O}_3$  solid solutions.
- $\text{Al}_2\text{O}_3$  takes significant amounts of  $\text{Nb}_2\text{O}_5$  into solution, but well below 10%; again the phase-boundary recedes with temperature.

The structure and other characteristics of the aluminium niobate  $\text{AlNbO}_4$  does not appear to have been determined yet. Its existence was, however, mentioned by Brandt<sup>12</sup> and earlier workers. An aluminium tantalate is known, but upon preparation, its structure was found to differ from that of the present niobate.

A schematic diagram is shown in Fig. 1o.

#### System $\text{Nb}_2\text{O}_5$ — $\text{SiO}_2$

In the system niobium pentoxide-silica, a series of fused mixtures prepared showed that  $\beta$ - $\text{Nb}_2\text{O}_5$  throughout coexisted directly with  $\text{SiO}_2$ , and that no devitrification occurred even after extended annealing at 800° C. (not of course barring an eventual one at higher temperatures). However, a significant amount of  $\text{SiO}_2$  enters into solid solution in  $\text{Nb}_2\text{O}_5$ , as again revealed by minor

TABLE XIV.—SYSTEM  $\text{Nb}_2\text{O}_5$ — $\text{Al}_2\text{O}_3$ 

| Composition (Molecular % $\text{Al}_2\text{O}_3$ ) | Heat Treatment   | Constitution  | Remarks   |
|--|------------------|---|---|
| 10%  | As melted        | $\beta$ - $\text{Nb}_2\text{O}_5$ solid solution, pure                                    | Same slight structural distortion as for $\text{TiO}_2$ |
| 10%  | 800° C., 14 days | $\beta$ , large<br>Phase $I_{Al}$ , small   |   |
| 25%  | As melted        | $\beta$ , large<br>$I_{Al}$ , medium  | $I_{Al}$ = compound $\text{AlNbO}_4$                    |
| 25%  | 800° C., 14 days | $\beta$ , large/medium<br>$I_{Al}$ , large/medium   |   |
| 50%  | As melted        | $\beta$ , small/medium<br>$I_{Al}$ , large/medium<br>$\text{Al}_2\text{O}_3$ , small      |   |
| 50%  | 800° C., 14 days | $I_{Al}$ , almost pure<br>$\beta$ , trace, if any   |   |
| 75%  | As melted        | $I_{Al}$ , large/medium<br>$\text{Al}_2\text{O}_3$ , small/medium                         |   |
| 75%  | 800° C., 14 days | $I_{Al}$ , large/medium<br>$\text{Al}_2\text{O}_3$ , small/medium                         |   |
| 90%  | As melted        | $\text{Al}_2\text{O}_3$ , large/medium<br>$I_{Al}$ , medium                               |   |
| 90%  | 800° C., 14 days | $\text{Al}_2\text{O}_3$ , large/medium<br>$I_{Al}$ , medium, but increased from as-melted |   |

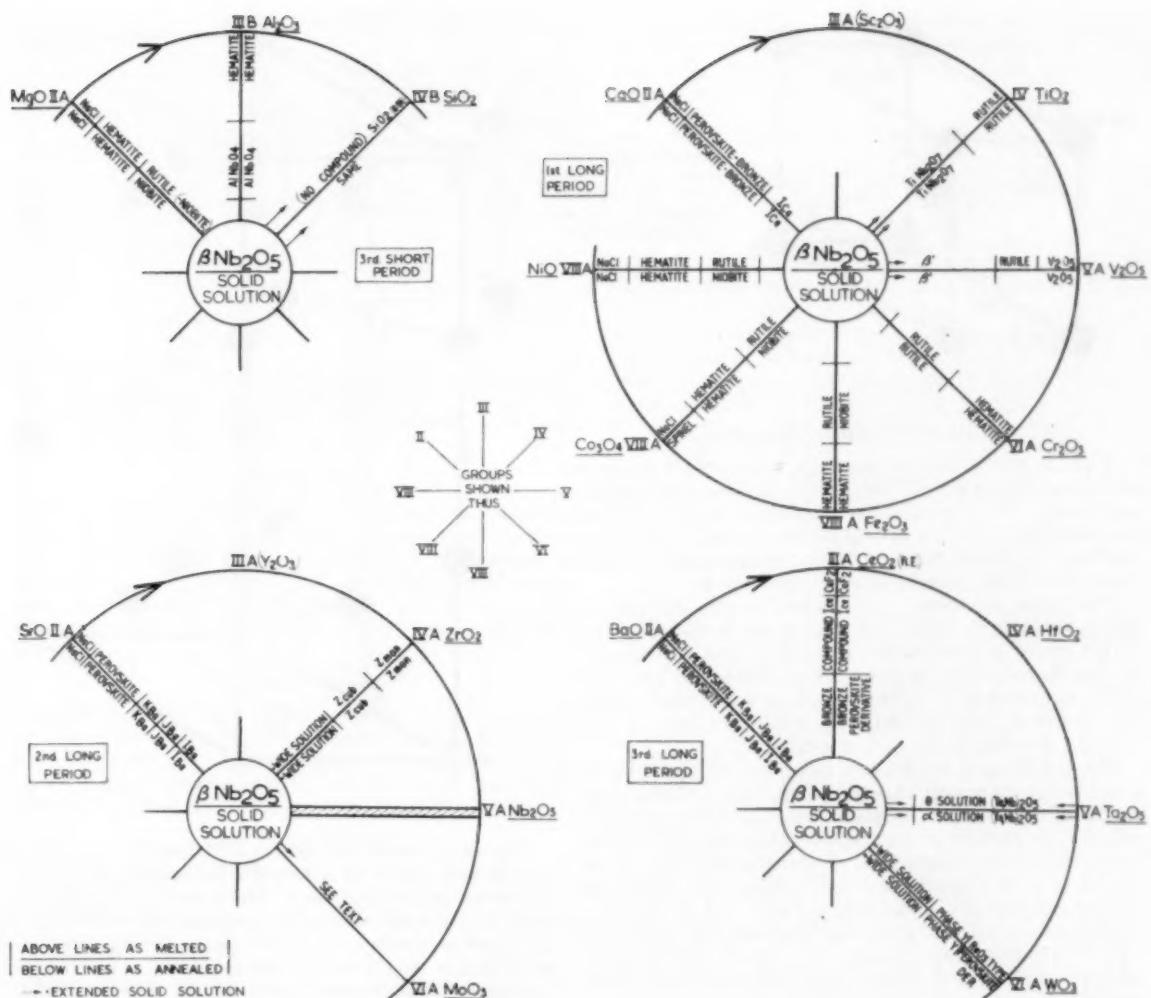


Fig. 4.—Survey of structures occurring in systems  $\text{Nb}_2\text{O}_5$ —second oxide, as a function of the periodic table.

modifications in the diffraction pattern. A tendency exists to stabilise  $\alpha\text{-Nb}_2\text{O}_5$  towards higher temperatures, as referred to previously.<sup>1</sup> No intermediate phase was found in this system.

#### DISCUSSION AND GENERAL CONCLUSIONS

A survey of the results from the aspect of crystal chemistry brings out many features which cannot all be discussed here. In the light of the periodic table, the constitutions may be shown as in Fig. 4. The broad pattern emerging is as follows:—

##### Solid Solutions in $\beta\text{-Nb}_2\text{O}_5$

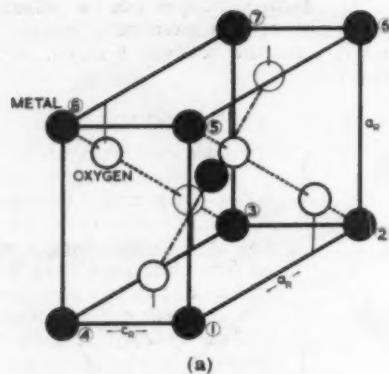
All the added oxides possess an appreciable, some a very extensive, solubility in  $\text{Nb}_2\text{O}_5$ , with a tendency to minor distortions in, but essential maintenance of, the  $\beta$  structure. This flexibility is particularly marked for the oxides of the transition elements adjoining Nb in

Groups IV, V and VI (Ti, Zr, V, Ta, Mo, W), each of which causes within  $\beta\text{-Nb}_2\text{O}_5$  characteristic variations in structure, spacing and stoichiometry. Fig. 2 shows some of the changes in diffraction pattern involved.

The  $\beta$  solubility generally decreases with decreasing temperature (a fact believed of interest as implying the possibility of precipitation effects within a scale-layer or bulk ceramic of this type, and of lattice stresses during nucleation of the next higher compound).

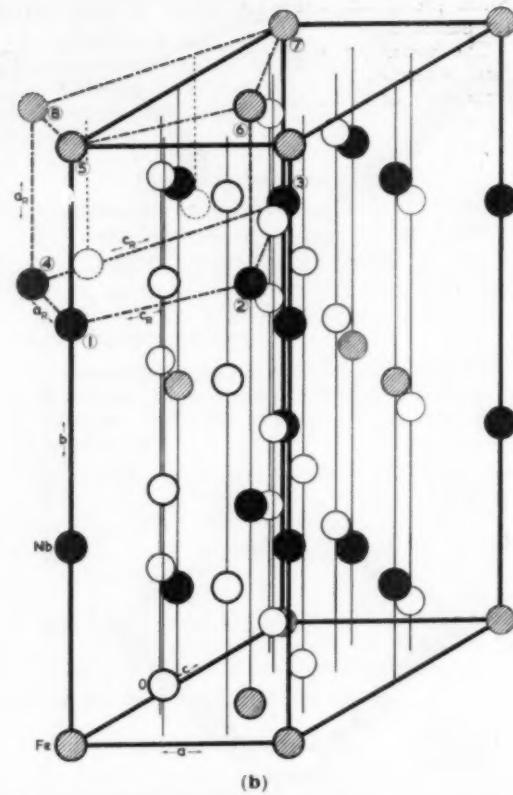
The new phase  $\beta'\text{-(Nb, V)}_2\text{O}_5$  occurring for vanadium addition is distinctive, and parallels the titanate  $\text{Nb}_2\text{TiO}_4$ , but, as the latter, is also considered a subsidiary variant to  $\beta\text{-Nb}_2\text{O}_5$  only. The solubility of  $\text{WO}_3$  in  $\text{Nb}_2\text{O}_5$  is the most extensive and of some added importance in the light of the diagonal relationship  $\text{Ti} \rightarrow \text{Nb} \rightarrow \text{W}$  and in creating an adaptable oxide lattice of type and range  $\beta\text{-(Nb, Ti, W)}_{2-3}\text{O}_5$ .

The only oxide which, according to present evidence, truly stabilises  $\alpha\text{-Nb}_2\text{O}_5$  is  $\text{Ta}_2\text{O}_5$ .



(a)

Fig. 5.—Rutile (a) and columbite (b) unit cells and atom movements required during transformation.



(b)

### Rutile/Columbite (Niobite)

A phase of rutile-type structure is stabilised through additions of Group VIII (Ni, Co, Fe) oxides and through magnesia. These rutiles are, however, stable at high temperatures only, and transform to the columbite (niobite)-type structure at lower temperatures. This rutile/columbite transformation is reversible.

The rutile structure also occurs in the systems of  $\text{Nb}_2\text{O}_5$  with  $\text{Cr}_2\text{O}_3$ ,  $\text{V}_2\text{O}_5$  and (obviously as end-member)  $\text{TiO}_2$ ; in these cases rutile exists alone and no transformation to columbite occurs. Throughout,  $\beta\text{-Nb}_2\text{O}_5$  is in direct equilibrium with rutile and/or columbite; these are therefore the potential precipitants out of the  $\beta\text{-Nb}_2\text{O}_5$  solution at lower temperatures.

The rutiles and columbites are sufficiently similar in composition to permit this transformation without long-range diffusion. Figs. 5a and b show the tetragonal unit cell of rutile and the larger orthorhombic cell of columbite, from which the mechanism of transformation may be traced; a rutile-equivalent cell within the columbite can be outlined, as shown, but a major change of shape of this pseudo-cell and shift of atoms is clearly involved.\* The octahedral environment of oxygen around metal atoms is maintained, with a slight distortion, during the transformation.

The ilmeno-rutile and tapiolite variants are comprised by the present rutiles, as crystallographically they do not essentially differ. Varying degrees of ordering are, however, possible, which may well affect the rate of transformation to columbite. The structural work of Karin Brandt,<sup>12</sup> Sturdvant,<sup>13</sup> Byström *et al.*<sup>14</sup> and Legrand and Delville<sup>15</sup> on rutile and columbite should be referred to here.

A major feature of the present rutiles and columbites is their wide deviation in composition from stoichiometry (nominal  $\text{AO}_2$  or  $\text{ABO}_4$ ). The extensive homogeneity ranges for the two structures within the binary systems (see Fig. 1) are evidence for this. The composition bands do not necessarily coincide for the two types, but they overlap. Variations in lattice dimensions, as recorded above, accompany these composition changes, and occur both within and between the systems.

There is little doubt that manganese (Group VII) niobates also include a rutile, though this system has not here been studied; whether or not transformation to a columbite occurs may be a very critical question, seeing that it does for  $\text{Fe}_2\text{O}_3$ , but not for  $\text{Cr}_2\text{O}_3$ .

\* An actual space-model which has been built in the author's laboratory demonstrates this process more plainly.

The observation of the rutile/columbite transformation is believed to be a new finding (and perhaps one of the most significant ones). The practical implication is that, for example, the volume changes and defect lattices involved are liable to affect the physical properties (such as adhesion and diffusion resistance) of a surface scale containing these phases, and to render them controllable by thermal treatment.† Similarly those of bulk ceramics would be affected.

The existence of several of the present columbite compositions is believed not to have been reported before, as is that of the (Nb, Mg) rutile.

The continuous rutile solution  $\text{TiO}_2 \rightarrow \text{NbO}_2$  observed (Fig. 3) would suggest ready rutile stabilisation in place of  $\text{Nb}_2\text{O}_5$  in scales on some Nb-base alloys, under conditions of restricted oxygen access (or under reducing conditions) entailing no doubt a major influence upon scaling behaviour.

### Hematite, Spinel and Rocksalt Structures

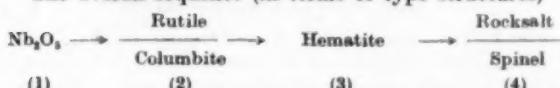
Hematite-type niobates follow on to the rutiles in several systems, namely those with Ni, Co, Fe, Cr and Mg. For Fe and Cr they obviously form the terminal phases ( $\text{Fe}_2\text{O}_3$ ,  $\text{Cr}_2\text{O}_3$ ); these are able to take considerable amounts of  $\text{Nb}_2\text{O}_5$  into solution, again with a temperature recession, implying that Nb-saturated hematites

† In mineralogy the rutile  $\rightarrow$  columbite change would be of interest, in so far as columbite is, of course, one of the main sources of niobium; it could, in the above light, be regarded as a product of transformation (in geological times) from high-temperature rutiles, in which form the mineral may still be liable also to occur. It seems likely then that the co-occurrence in a deposit of a rutile with a columbite would label the former as a parent, and as of similar composition to the latter, i.e. equally high in niobium (or tantalum).

could precipitate rutile or columbite with decreasing temperature.

The hematites in the  $\text{NiO}$ ,  $\text{Co}_3\text{O}_4$  and  $\text{MgO}$  systems form intermediate phases, followed finally by the  $\text{NaCl}$  structures, or (in the case of cobalt at low temperatures), by spinel. All hematites are stable at high and low temperatures; they undergo changes in lattice-dimensions as shown by the figures quoted, and possess a pronounced degree of non-stoichiometry relative to the  $\text{Me}_2\text{O}_3$  compositions.

The overall sequence (in terms of type structures)



is thus of general validity in this group of systems with the proviso that the allotropy may or may not be omitted, and that the series may terminate already at stage (3) (for  $\text{TiO}_2$  at stage (2)).

### Zirconia

$\text{Nb}_2\text{O}_5$  tends to stabilise the cubic (high temperature) form of  $\text{ZrO}_2$ . The  $\text{Nb}_2\text{O}_5$ - $\text{ZrO}_2$  system therefore gives the relatively simple overall picture of containing, as only phases, the primary  $\text{Nb}_2\text{O}_5$  and  $\text{ZrO}_2$  modifications without new intermediate niobates; this it has significantly in common with  $\text{Nb}_2\text{O}_5$ - $\text{TiO}_2$  (presumably also  $\text{HfO}_2$ ).

### Tungsten Oxide

Apart from the wide  $\beta$ - $\text{Nb}_2\text{O}_5$  solution of  $\text{WO}_3$  referred to, the intermediate niobate  $\text{Y}$  (approximately  $\text{Nb}_2\text{W}_2\text{O}_8$ ) appears new and worth further investigation. The small primary solution in  $\text{WO}_3$  leads on to the perovskite group structures (see below).

### The $\text{Ta}_2\text{O}_5$ Forms

The existence of the high temperature form of  $\text{Ta}_2\text{O}_5$  already reported,<sup>7</sup> but later denied,<sup>8</sup> is here certainly confirmed. The present wide solid solubility of  $\text{Nb}_2\text{O}_5$  in both the  $\alpha$  and  $\theta$   $(\text{Ta}, \text{Nb})_2\text{O}_5$  forms (up to at least 60%  $\text{Nb}_2\text{O}_5$ ) is additional information. The  $\alpha \rightarrow \theta$  transformation is reversible (in contrast to the  $\alpha \rightarrow \beta$ - $\text{Nb}_2\text{O}_5$  change); it appears, for pure  $\text{Ta}_2\text{O}_5$ , between

1,400 and 1,450°C. Both allotropes can be caused to coexist metastably, and certain interesting transitional stages are found in structure between  $\theta$  and  $\alpha$ , which invite further study.

### The Perovskite Derivatives

The perovskite type of structure appears prominently in the systems of  $\text{Nb}_2\text{O}_5$  with the alkaline earth oxides ( $\text{CaO}$ ,  $\text{SrO}$  and  $\text{BaO}$ ), as well as with rare earth oxides ( $\text{CeO}_2$  base). The phases possess appreciable homogeneity ranges, accompanied by spacing changes with  $\text{Nb}_2\text{O}_5$ /second oxide ratio. The unit cells vary from cubic to tetragonal (see data in tables), with some possible further lowering to orthorhombic symmetry. There occur, in at least two of the systems, phases of the tungsten bronze type of structure ("tetragonal bronzes"), closely related to the perovskites.  $\text{Na}_x\text{WO}_3$  ( $X < 1$ ) is the prototype. The subtractive lattice, by which the bronzes are characterised, becomes, for  $X = 1$ , the ideal perovskite ( $\text{ABO}_3$ , e.g.  $\text{CaTiO}_3$ ), and for  $X = 0$  the rhenium oxide ( $\text{ReO}_3$ ) structure, to which  $\text{WO}_3$  is also related. The two terminal structures are represented in Fig. 6, showing the simple perovskite and  $\text{ReO}_3$  unit cells, where the essential difference is the presence or absence of the central large metal atom. The partial occupation of this site typifies the bronzes and would be associated with various degrees of minor distortion. (The work of Magnéli and *et al.*,<sup>16</sup> Hägg,<sup>17</sup> Roy,<sup>18</sup> Geller,<sup>19</sup> Rosen *et al.*,<sup>20</sup> Rooksby<sup>21</sup> and Straumanis<sup>22</sup> on the perovskites and bronzes should here be referred to, as well as the basic structure studies of Megaw<sup>23</sup>.)

In this case, we are dealing with niobium bronzes, which form what is probably a continuous replacement series of solutions with the perovskites. Niobium bronzes and perovskites are known already from Ridgley and Ward's<sup>11</sup> and Krylov's<sup>9</sup> work. The alkali niobates ( $\text{NaNbO}_3$ ,  $\text{KNbO}_3$ , e.g. Megaw and Wells,<sup>24</sup> Wood<sup>25</sup>) are here of additional interest; they have also been synthesised and examined in the present work for comparison with the other perovskites. They would logically fall into position on our circular representation in Fig. 4 at the left. The marked structural variations in the new (as in the known) perovskites may incidentally also

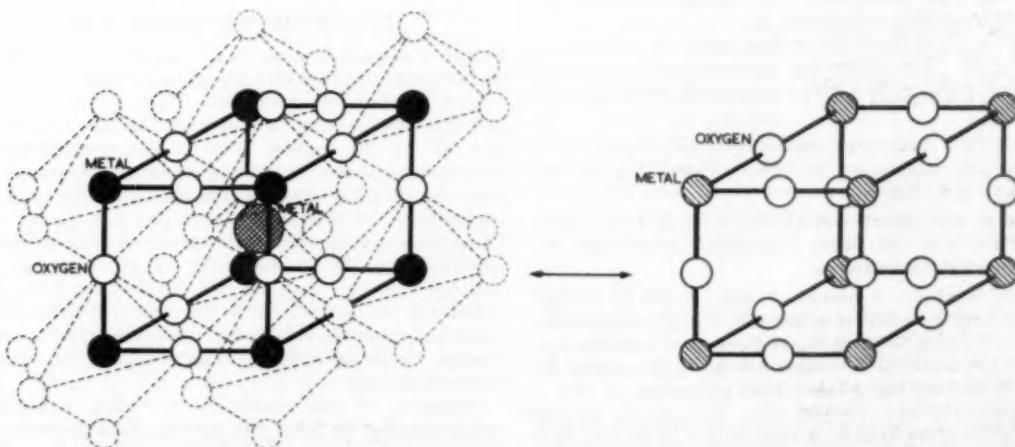


Fig. 6.—The unit cells of perovskite type and rhenium oxide type niobium bronze through intermediate occupation.

make them of some interest from the semiconductor side.

There seems little doubt that the niobium perovskites will also include scandium, ytterbium and lanthanum compounds (Group III A, intervening between the Ca and Ti Groups and leading to the rare earths, where they were verified for  $\text{CeO}_2$ ). On the other side of the niobium block, rhenium in Group VII, like tungsten, is clearly also a stabiliser of the perovskite structure, of the  $\text{ReO}_3$  variant, in which niobium could enter (cf. Fig. 6). A governing structural theme is throughout that of the  $\text{MeO}_6$  octahedra common to the bronzes and their perovskite and  $\text{ReO}_3$  type termini.

In the alkaline earth systems, perovskite occurs in direct equilibrium with primary  $\text{CaO}$ ,  $\text{SrO}$  and  $\text{BaO}$ . The latter themselves dissolve only minor amounts of  $\text{Nb}_2\text{O}_5$ , which contrasts with the rare earths, where  $\text{CeO}_2$  takes considerable amounts of  $\text{Nb}_2\text{O}_5$  into solution.\*

#### Other Compounds

The niobates  $\text{I}_{\text{Ca}}$ ,  $\text{I}_{\text{Ba}}$ ,  $\text{I}_{\text{Sr}}$ ,  $\text{J}_{\text{Ba}}$ ,  $\text{J}_{\text{Sr}}$  and  $\text{K}_{\text{Ba}}$ ,  $\text{K}_{\text{Sr}}$ , intermediate to  $\beta\text{-Nb}_2\text{O}_5$  and the perovskite types are here only noted and have not been further investigated; the same applies to phase  $\text{I}_{\text{Ce}}$  between perovskite and  $\text{CeO}_2$ . The aluminium niobate  $\text{NbAlO}_4$  ( $\text{I}_{\text{Al}}$ ) observed is known,<sup>12</sup> but (a) its appreciable homogeneity range and (b) its coexistence directly with  $\beta\text{-Nb}_2\text{O}_5$  and  $\text{Al}_2\text{O}_3$  are recorded.

#### Melting Points

Melting points within many of the systems have been measured, but will be reported at a later stage. Finally,

\* The simple picture thus arises that the rocksalt type lattice of  $\text{CaO}$ , etc., transforms almost immediately to perovskite, upon  $\text{Nb}_2\text{O}_5$  addition, but the fluorite lattice of  $\text{CeO}_2$  is much more accommodating, before, first, a new phase and only then the perovskite is formed. A closer comparison of the three structures ( $\text{NaCl}$ ,  $\text{CaF}_2$ ,  $\text{CaTiO}_3$  types) is available metal and oxygen sites and electron distributions proves instructive in suggesting the reasons and the scope for non-stoichiometry; however, this forms a separate subject, which cannot be enlarged on here.

the above work should only be considered as exploratory, but a consistent and systematic pattern already emerges, in which primary  $\text{Nb}_2\text{O}_5$  solid solutions of varying extent and type, and compounds of rutile, columbite, hematite, perovskite and other types of structure figure prominently. Several of the phases and their transformations are new, and believed to be of some interest and application in wider fields. Varying homogeneity ranges and degrees of non-stoichiometry of these phases are a frequent feature.

#### ACKNOWLEDGMENTS

The author expresses his sincere thanks to Mr. D. A. Oliver, C.B.E., Director of Research, The B.S.A. Group Research Centre, for his encouragement, and to Messrs. K. Albutt and M. D. Eccleston for their assistance in the experimental work. He is indebted to Mr. H. P. Rooksby and Dr. M. K. Francombe (lately) of the G.E.C. Research Laboratory, Wembley, for a helpful discussion and loan of three mineral samples.

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#### ADDENDUM

A paper† has recently appeared by Sandoz of the U.S. Naval Research Laboratory, Washington, reporting the successful use of zinc coating upon niobium in affording protection against high temperature oxidation. The effect consists in the formation of a combined zinc-niobium oxide upon the surface (its nature was not stated), which is self-healing and intergrown with an  $\text{Nb-Zn}$  intermetallic compound.

This links in a most interesting manner on to the present work. The writer has prepared and examined  $\text{Nb}_2\text{O}_5\text{-ZnO}$  melts in line with the present methods, and found:—

- (a) That the compound  $\text{ZnO}\text{-Nb}_2\text{O}_5$  ( $\text{ZnNb}_2\text{O}_6$ ) is a columbite similar to the others found, e.g. in the present  $\text{NiO}\text{-Nb}_2\text{O}_5$ .
- (b) That it is in direct equilibrium with  $\beta\text{-Nb}_2\text{O}_5$  solid solution (i.e. no other compound intervenes, as in the other columbites).
- (c) That, however, it occurs already in the as-melted condition, in contrast to our (Fe, Co, Ni) columbites above, which were lower-temperature transformation products from rutile, but to some extent in common with the present  $\text{MgO}$  columbite.

Seeing that in our periodic table systematics copper oxide follows on to  $\text{NiO}$ , and is followed by  $\text{ZnO}$  in the first long period, it was also of interest to complete the

link-up and prepare  $\text{Nb}_2\text{O}_5\text{-CuO}$  melts. This was done, with the result that a columbite type compound again appears, at  $\text{CuO}\text{-Nb}_2\text{O}_5$ . It too directly coexists with  $\beta\text{-Nb}_2\text{O}_5$  solid solution, and occurs already as melted, but in a structurally slightly modified form, possibly a transitory stage from rutile.

#### Electrodeposition of Tin

THE 10th annual Journée de l'Etain was held in Brussels on November 8th at the premises of Fabrimetal. The subject for the conference was "The Electrodeposition and the Applications of Tin and Tin Alloy Coatings," and Mr. R. M. Angles, head of the electrodeposition laboratories of the Tin Research Institute, presented a paper on tin-nickel and tin-zinc alloy coatings and their applications. Other papers included one by Mr. C. J. Thwaites, assistant chief metallurgist of the Tin Research Institute, on the solderability of tin and tin alloy coatings; one by M. J. Browet, head of the laboratories of Sabena, on tin-lead electrodeposits and their applications; and one by M. Riesser, of the Société Minerve de France, Paris, on tin alloy electrodeposits in the electronics industry.

Delegates to the conference included metallurgists and technologists from the leading French firms using solders, including representatives of the automobile industry, canned foods, radio and television and manufacturers of electronic equipment.

† Sandoz, G.: *Jnl. of Metals*, **12**, 1960 (April), p. 340.

# Developments at Alcan's Rogerstone Plant

## New Features of Hot and Cold Strip Lines

*Continued from page 206 of the November issue.*

### AUXILIARY EQUIPMENT

**G**AMMA-RAY gauge equipment, two ingot tilters, a slab shear and plate handling equipment are positioned between the 144 in. mill and the 96 in. mill approach tables. The gamma-ray gauge is mounted on a swivel stand at the side of the outgoing tables and relays the gauge of the metal being rolled to a recorder in the 144 in. mill control cabin. Screwdown adjustment can be carried out, in conjunction with this equipment, during rolling, the screwdown being designed for future operation with automatic gauge control.

Two three-prong ingot tilters of 6,000 lb. capacity are provided on either side of the slab shear. Hydraulically operated, they receive ingots vertically and lower them through 90° on to the tables. The tilter nearest the mill will receive ingots from the tunnel preheating furnaces, whilst the plate that has just been rolled is being sheared and lifted from the table. The other tilter, on the run-out table from the shear, will take ingots which are to be rolled by the 96 in. mill when the 144 in. mill is rolling plate.

### 144 in. Slab Shear

This is a vertical down-and-up-cut shear made by Davy and United Engineering Co., Ltd. It has a capacity of  $1.25 \times 10^6$  lb. and is designed to shear hot slab and plate having a maximum cross-sectional area of 300 sq. in. at temperatures between 400° C. and 500° C. The blades, 146 in. long with a rake of  $\frac{1}{2}$  in., are driven by two 400 h.p. A.E.I. low-inertia motors, fitted with a 30 in. Igranic type S.M. brake. Cutting speed is 8 or 16 cuts per minute at half or full power, respectively. The shear is fitted with hydraulic arrester gear, operating at 1,500 lb./sq. in., to hold the plate and prevent marking during the cutting operation. It is also provided with hydraulically-operated guides on the entry side to square up and centre long plates or slab before shearing. An underground conveyor carries the sheared ends to a crop shear, which cuts them to a manageable size; they are then lifted and deposited in scrap bins at floor level. The crop shear is powered by a 40 h.p. motor.

### Plate Transfer

A system of transfer beams, fitted over the tables immediately after the shear, removes finished slab or plate to the ingot storage bay, or loads it on to a plate car for eventual transfer to the plate stretcher and processing department.

### Lubrication—144 in. Mill and Auxiliaries

Lubrication systems for the 144 in. mill and auxiliary equipment are located in a 25 ft. deep oil cellar, on the outgoing side of the roll change side of the mill, and are as follows:—

- (1) The table system consisting of a 7,300 gallon settling tank from which two Mirrlees positive-

displacement pumps (one a stand-by) deliver at 225 gal./min., through a fully automatic Auto-Klean T.K. strainer and a pressure vessel, to all the mill tables.

- (2) The screwdown and screwbox systems have a common 1,800 gallon settling tank. Two Mirrlees positive-displacement pumps (one a stand-by) deliver oil to the screwdown at 45 gal./min. through a fully automatic Auto-Klean T.K. strainer and a pressure vessel. A further two Mirrlees pumps (one a stand-by) deliver to the screwbox at 5 gal./min., through a manual Auto-Klean strainer only.
- (3) The slab shear system consists of a 960 gallon settling tank from which two Mirrlees positive displacement pumps (one a stand-by) deliver at 30 gal./min., through a fully automatic Auto-Klean T.K. strainer and a pressure vessel.
- (4) The soluble oil lubrication system for centre bearings consists of an 800 gallon tank from which two size 10 U.L. LaLabour pumps (one a stand-by) deliver to the centre bearings on the turning tables and to the ingot tilters at 40 gal./min., through a manually operated Auto-Klean strainer.

Further lubrication is catered for by Farvel D.C.4 and D.C.25 units, both of which are sited at mill floor level. Roll necks are lubricated by grease (hand make-up) and oil mist from a unit on the screwdown platform.

### Hot Mill Handling

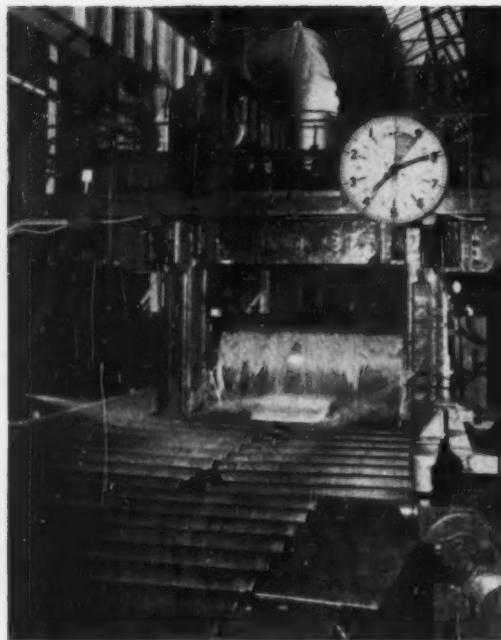
The 318 ft. long mill bay is traversed by a Vaughan 125 ton (25 ton auxiliary) E.O.T. crane. It is used for plate handling, roll-change operations and general maintenance work, and was used for the installation of the 144 in. mill stands and motors and general erection work in this bay. Access to the enclosed motor room is gained through high-level aluminium roller shutters.

### Roll Grinding

A new Waldrich roll-grinding machine has been installed at the southern end of the mill bay and is designed primarily for the 144 in. mill rolls, which may be ground in their chocks. It can also be used to augment existing roll grinders, and will accept any of the various rolls in the Rogerstone Mill. Power is provided by a 44 h.p. A.E.G. motor, continuously rated at between 400 and 1,300 r.p.m., which is coupled through a gearbox. The roll grinder foundations are set on anti-vibration steel springs and have hydraulic damping units.

### Outgoing Tables

The ingoing feed and turning tables are duplicated on the outgoing side of the mill. A further fifty-four rollers, 12 ft. wide and pitched generally at 36 in. centres, link the 144 in. mill with the 96 in. mill approach tables, which are 87 in. wide. The latter tables can be used as a stand-by for off-loading plate wider than 87 in. because the rolls stand proud of the table side frames.



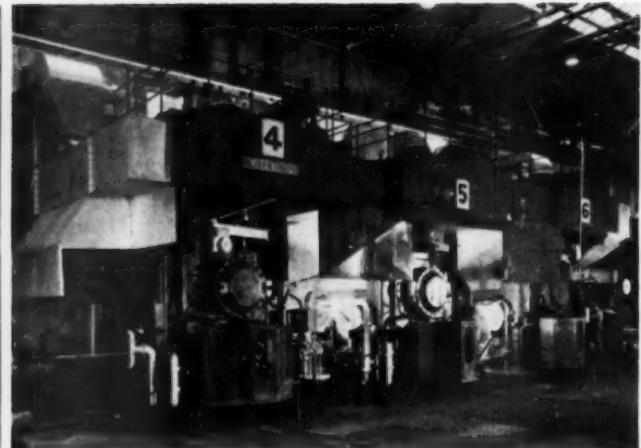
The Brightside 96 in. mill may be used for breaking down ingot or as an intermediate hot mill.

### The Hot Line

The addition of the new 144 in. mill and the provision of a third stand in the 88 in. hot finishing mill has led to further re-organisation of the line to preserve the continuity. When the 144 in. mill is working in conjunction with the hot line, the 96 in. mill acts as an intermediate mill. With this sequence the 84 in. intermediate mill is not needed, and it is therefore to be dismantled. On the occasions when the hot line is operating independently of the 144 in. mill, the 96 in. mill acts as the breakdown mill, and takes three to five extra passes and delivers slab direct to the hot-finishing mill.

The 96 in. mill, when acting as intermediate, handles far longer slabs than it was designed for. To assist the existing side guides in squaring up the slab, those associated with the 84 in. mill are to be relocated outside the existing guides. This stand will then be equipped with the full range of turning, centering and guiding equipment appropriate to a stand which has to break down ingots and also roll slab in long lengths.

The up-cut shear and edge trimmer, positioned immediately in front of the 88 in. finishing mill, will be moved to take the place of the 84 in. mill. The shear can handle 1 in. thick hot aluminium plate up to 80 in. wide and is driven by a 50 h.p. continuously-rated motor. Off-cuts from the shear fall down a chute to a conveyor, which lifts and deposits them in scrap bins at floor level. The shear is provided with side-guiding equipment on the entry side to deal with long slabs. The modernised edge trimmer can cut strip 0.6 in. thick and up to 72 in. wide. It is driven by a 120 h.p. motor, which is controlled to synchronise the cutter speed with that of the table. In its new position it will edge trim slabs up to 200 ft. long without interfering with other operations



Hot finishing is carried out in a single pass on this Robertson 88 in. three-stand tandem mill.

of the line. Existing controls for the shear and trimmer will be relocated in the present 84 in. mill control cabin.

### 88 in. HOT FINISHING MILL

The new stand in the tandem hot-finishing mill is set in front of the two existing stands on foundations laid when the line was assembled in 1949-50. Made by W. H. A. Robertson & Co., Ltd., it is identical to those originally installed. It is an 88 in. wide, 4-high non-reversing mill, with 24 in. diameter work rolls and 56 in. diameter back-up rolls. The centres of the rolls are 19 ft. in front of the centre of the following mill. The mill housings are made of cast steel and weigh 90 tons. Roll journal bearings are of the Robertson flood-lubricated type, consisting of white metal sleeves which are mounted in cast steel chocks and located axially by ball-races. The work-roll chocks are held between horns on the back-up-roll chocks by wedges, which can be used to offset the work rolls by as much as  $\frac{1}{2}$  in. The rolls are of forged steel, 70° Shore hardness in the case of the work rolls and 50°/55° Shore hardness for the back-up rolls.

### Screwdown

The screwdown system is driven at 0.25 in./min. by two 50 h.p. D.C. motors through worm reduction gears. A magnetic clutch, linking the drives, is released for differential adjustment. The maximum gap obtainable between work rolls is 6 in.

### 88 in. Mill Drive

The new stand is driven, at 150/300 r.p.m., through a gearbox made by David Brown Industries, Ltd., by a 4,000 h.p. A.E.I. single-armature D.C. motor and is coupled to a 1/1 pinion box by a Holset coupling. Gearbox reduction is 3.75 : 1. The centre stand, originally the entry stand, was powered by a 3,000 h.p. motor through a slab coupling and 60 in. face pinions; the motor has been replaced by a second 4,000 h.p. A.E.I. single-armature D.C. motor, operating at 58/116 r.p.m., with a direct drive to the mill pinion box. The 2,600 h.p. motor on the exit stand, driving an existing gearbox and 1/1 pinion stand, is retained.

### *Roll Coolant*

A 10% Sternol/soft-water emulsion is sprayed through separately-controlled banks of jets over the section of the rolls in contact with the strip. The original system, with a 10,000 gallon dirty-oil tank and a forced draught cooler, was designed for four stands and has required no major modification, but two new 90 gal./min. Pulsometer pumps have been installed and the pumphouse has been rearranged to improve the efficiency of the system.

### *Fume Extraction*

With the installation of a third stand, it has been necessary to increase the fume extraction capacity by 20,000 cu. ft./min. to a total of 80,000 cu. ft./min. A new oil eliminator, similar to the one on the 144 in. mill system, has been installed. All ducting is constructed in aluminium.

### *Lubrication*

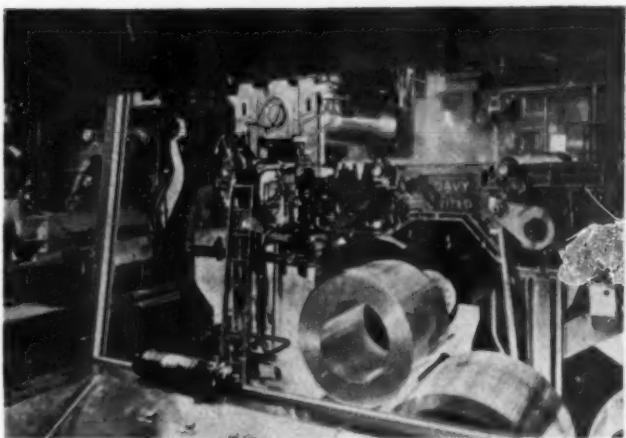
The original lubrication system was designed for four stands, and is based on two 2,000 gallon settling tanks, from which oil is pumped for the flood lubrication of the gear bearings and the spraying of the main drive gears.

### *88 in. Mill Capacity*

The modified tandem finishing mill can now process soft and hard alloys at rolling speeds of up to 800 ft./min. Slabs will be delivered to the mill in thicknesses usually between 1.000 in. and 0.500 in. Strip can be rolled in the mill to any gauge between 0.080 in. and 0.25 in. and coiled. Normal finishing gauges will be between 0.100 in. and 0.140 in.

### **TRIMMING AND COILING**

In the rolling sequence of the original hot line, the 88 in. mill was followed by a 651 ft. table fitted with cooling sprays to reduce the strip to a temperature at which it could be satisfactorily coiled. At the end of the table the strip was edge-trimmed and then up-coiled.



Seen through the pulpit window, a hot coil is ejected from the down-coiler.

Developments in hot coiling since the mill was first laid down have made it possible to coil strip satisfactorily without first having to cool it. New edge trimming and coiling equipment has therefore been fitted immediately following the last stand of the finishing mill.

### *Edge Trimmer*

The edge trimmer, made by Stamec, can accept strip at 200°-300° C., in both common and strong alloys, between 24 in. and 80 in. wide. Strip up to 0.25 in. thick can be handled, trimming speeds varying between 800 and 450 ft./min. according to gauge. The pinch rolls and cutters are driven by a 150 h.p. motor, the drive being synchronised with the mill speed. Scrap is cut to manageable lengths by another cutter, evacuated by a conveyor system under the tables, and then deposited in scrap bins set at floor level.

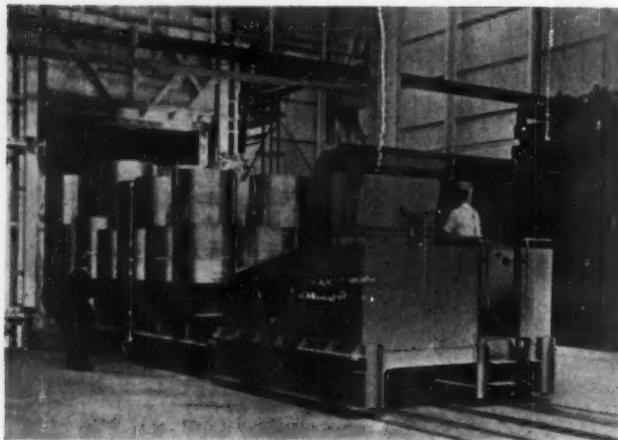
### *Hot Coiler*

The centre-line of the reel of the mandrel down-coiler, which was built by Davy and United Engineering Co., Ltd., is set 25 ft. 6 in. from the last stand of the 88 in. mill. It has a two-speed gearbox and will coil strip between 0.080 in. and 0.25 in. thick, 28 in. to 80 in. wide, and at temperature between 200° C. and 300° C., at 450 or 800 ft./min., matching the speed of the edge trimmer. The coils, which have an inside diameter of 24 in. are limited to outside diameters of between 32 in. and 54 in. and a maximum weight of 9,000 lb.

Strip emerging from the edge trimmer is caught between pinch rolls, which divert it to the coiling reel. The upper pinch roll, 24 in. diameter, is driven through a 4.08 : 1 gear; the lower roll, 16 in. diameter, through a 2.86 : 1 gear. Common to both pinch rolls is a 35 h.p. motor. The strip is directed round the reel by a belt wrapper which is retained until there are sufficient turns to hold the rolling tension. The tension range of coiling through the gearbox varies from 9,000 lb. to 48,000 lb., for speeds of up to 450 ft./min., to 0-9,000 lb. for speeds of 450-800 ft./min. The reel, which is of the four-segment, collapsing type,



The hot-rolled coils are cold-rolled in this Davy-United 66 in. three-stand tandem mill.



Charging one of the new oil-fired annealing furnaces with a tray of coils.

concentric when open, is driven through a two-speed gearbox by a 400 h.p. A.E.I. motor with a speed range of 375 to 1,100 r.p.m.

On completion of coiling, the coils are removed from the collapsed mandrel by a hydraulically-operated stripper car to a conveyor. They are weighed while on the conveyor and are eventually lifted by crane and placed in a storage bay to cool.

Foundations have been laid for a second coiler which will be positioned at a distance of 12 ft. from the first coiler.

#### Cold Rolling

For cold rolling, the coils are transferred by crane to the existing Davy-United 66 in. three-stand tandem mill. The demand on this mill has been somewhat eased by improvement of the hot line which has made possible the production of larger coils in a greater range of thicknesses. To increase the existing cold-rolling capacity, however, a 72 in. two-stand tandem mill, extensively modernised, is being transferred from the East Works.

#### 72 in. TANDEM MILL

This is a Robertson two-stand, 4-high, non-reversing mill with rolls of 15 in. and 37 in. diameter. When the work on the mill is completed, it will be able to roll coils of heavier-gauge metal at greatly increased speeds. The modernisation of this mill is being carried out in four stages. The first, which has been completed with the mill in its present position, involved improvements to the roll coolant system to maintain a high standard of surface finish, and new feed arrangements, enabling the mill to handle coils down to 0.016 in. entry gauge, as compared with the original minimum of 0.050 in.

The mill will be moved to the new cold mill building in January, 1961, and will be fitted with new motors in the second stage of modernisation. These motors, of 2,200 h.p., will be fitted to each stand with new pinion stands, new bearings, and reel and belt wrapping equipment. When this stage is completed the

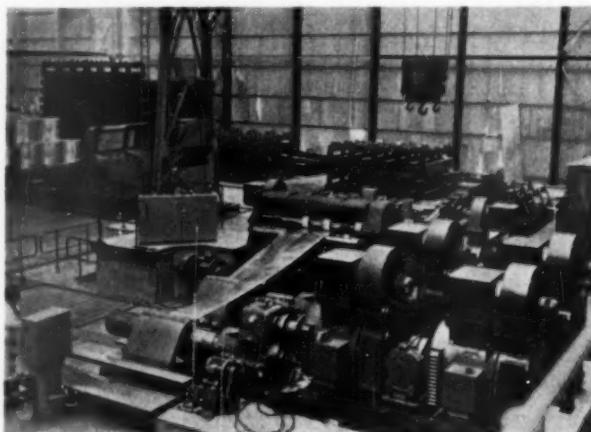
mill will be capable of rolling at 2,500 ft./min., compared with its present speed of 360 ft./min. The two final stages cover the full mechanisation of handling equipment and installation of control refinements, and of two further 2,200 h.p. motors, linked in tandem, giving a 4,400 h.p. drive to each stand and consequently greater torque for rolling thicker gauges.

#### ANNEALING FURNACES

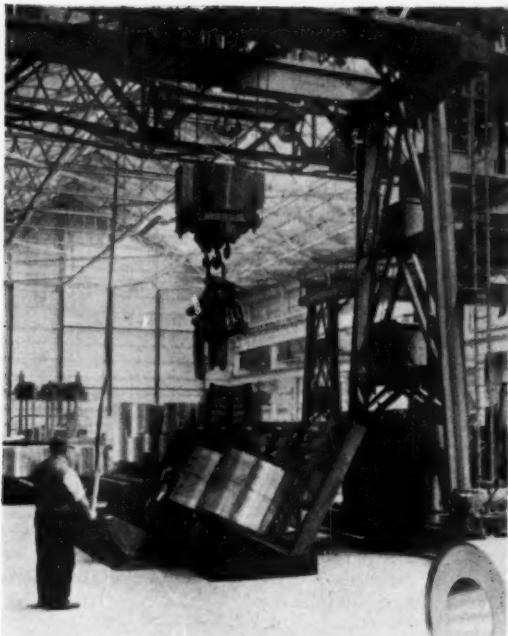
The greater amount of strong alloy in the increased output of the mill has required a more than proportional increase in annealing capacity. To this end, existing gas and electric furnaces have been augmented by two new Stein & Atkinson oil-fired coil-annealing furnaces, with controlled-atmosphere plants. They are located in an annexe to the new cold bay building.

The furnace and furnace-door construction is a steel shell, lined with heat-resisting alloy plates and Stillite insulation. The door is counterbalanced to assist the raising and lowering winch and, when closed, is pressed by hydraulic pushers against a seal of water-cooled sponge strip insert. Each furnace has a clear load space of 2,230 sq. ft., is 27 ft. long  $\times$  10 ft. high  $\times$  8 ft. 3 in. wide, and can accept sixteen coils of 46 in. outside diameter stacked 70 in. high, weighing a total of 67 tons. Different coil sizes would allow a possible maximum charge of 112 tons.

Special burners, developed by Stein & Atkinson in conjunction with Surface Combustion Corporation, use marine-diesel oil to heat high-quality stainless steel radiant tubes fitted in the sides of the furnace and insulated from the furnace interior. Air is drawn over banks of these tubes by water-cooled recirculating fans, supplied by Keith Blackman, Ltd., and is then ejected at the base of the furnace sides into plenum chambers zones, which may be separately controlled, and which are a part of the annealing tray. The hot air then passes up through the load and is recirculated by the fans. The fans, driven by 40 h.p. motors at 1,480 r.p.m., develop 1½ in. water gauge, giving a delivery of 30,000 cu. ft./min. at 600° C. The furnaces have an operational heating range from 150°–600° C. and can hold a selected



The new 72 in. slitting line in operation.



Part of the coil handling equipment in the cold mill bay.

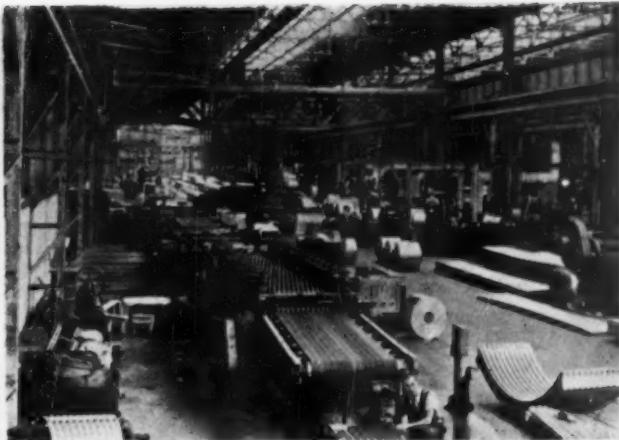
temperature within that range of  $\pm 3^{\circ}\text{C}$ . They operate nominally on a 12-hour cycle per load, which gives an annual throughput of between 24,000 and 30,000 tons.

Coils that are to be annealed are stacked, lying either vertically or horizontally, on specially-designed trays. The trays are handled by a 50 ton motorised grab and lifted on to a furnace bogie by a 60 ton Morris crane serving the new cold mill bay. The bogies are on rails opposite the furnaces and are charged or discharged by an electrically-driven cab unit.

A Holmes Kemp controlled atmosphere plant has been installed by Stordy Engineering, Ltd. This plant will give a non-oxidising atmosphere which reduces the amount of oil staining at low temperatures. Automatic control is used and has been supplied by Honeywell Controls, Ltd. The system may be preset to requirements, and incorporates all the usual indicators and recording equipment.

#### 72 IN. SLITTING LINE

A new high-speed slitting line, designed and built by Sir James Farmer Norton, Ltd., has been erected in the new cold bay extension to edge- and centre-slit strip between 0.008 in. and 0.065 in. thick, in both hard and soft alloys. The line can handle coils up to 8,000 lb. in weight, having an unfinished width limit of between 30 in. and 80 in. and a maximum outside diameter of 52 in. The slitting speed is 2,000 ft./min. A pneumatically-operated down-ender accepts coils from a loading crane and tilts them on to a roller conveyor. The coils are transferred to the ingoing coil carriage and there "tailed" by tailing rollers (i.e. the first few feet of the coil is straightened out) before being directed to the unwind cones. Otherwise conventional, the unwind cones have an automatic centering unit to



View of the cold mill bay with the corrugating line in the foreground.

correct misalignment of the coils, and are monitored by an air jet. Drag generators are fitted to the unwind cones to give a maximum back tension of 3,000 lb.

The strip is passed between ingoing feed rolls to the slitter, of standard Alcan Industries, Ltd. design, a twin-mandrel pull-through type with stationary arbors and rotating cutter housings. Scrap is cut to lengths of approximately 12 in. by a rotary flying-shear cutter, and delivered by chute and conveyor to scrap bins at floor level. Twin re-coiling reels are fitted, which will accept coils in widths between 36 in. and 78 in. The reels are driven by two variable-speed A.C. motors in tandem, only one being used when a half-coil is slit. The finished coil is lifted and transported on a carriage to an up-ender before being removed by crane.

The slitting line is controlled from an elevated platform alongside the slitter. From this position the operator can view the strip from the unwind cones to the coiling reels. There are three auxiliary control stations placed by the tailing mechanism, the unwind cones, and the reels.

#### HANDLING EQUIPMENT

A Morris 60 ton E.O.T. crane is provided in the new cold bay. The crane has drum-operated master controllers. The hoist and cross-traverse have dynamic braking and there is a foot-operated brake for long travel. When used for handling the annealing trays, the crane main hoist is fitted with a 50 ton motorised lifting grab made by Kings Hoists, Ltd., and controlled from the crane cabin. A motorised beam, also controlled from the cabin, has been supplied to handle four 9,000 lb. coils simultaneously when charging the existing G.W.B. furnaces, whose charging arrangements differ from those of the new furnaces. The crane will also be able to deal with roll changing on the 72 in. cold mill.

Two Morris 6 ton semi-goliath cranes will handle coils on either side of the new slitting line. The cranes have a span of 48 ft. 6 in. and a crane rail height of 22 ft. The 6 ton hoist is fitted with a rotatable triple hook, and has a hoist speed of 30 ft./min. and a cross-traverse of 100 ft./min. The cranes are controlled from the floor. Another 6 ton semi-goliath crane will work over a 6-coil

upender, and is similar to those described above, but has a shorter span of 29 ft. 6 in. and a crane-rail height of 20 ft. Two further semi-goliath cranes will be installed to operate either side of the 72 in. cold mill when it is relocated.

#### Finishing

After cold rolling, the coils are transferred to the finishing bays, where they pass down various production lines, according to whether they are required for corrugated building sheet, container sheet, or circles for hollowware. Foil stock normally needs no further treatment after the final anneal.

#### Heavy Shear Lines

Coils required for building sheet pass down the heavy shear lines, where they are unwound, flattened by a roller leveller, cut to length by a flying shear, and finally corrugated. Some profiles are made in roller machines and others in brake presses. To obtain a much favoured stucco finish the coils are given a separate rolling operation, using embossed rolls, before corrugating.

#### Light Shear Lines

Standard-sized container sheets are produced on the automatic light shear lines to which the coils are transferred after leaving the temper mill. Passing through the plant at 600 ft./min., they are unwound, degreased

to remove excess rolling oil, inspected visually for surface finish and mechanically by a flying micrometer for gauge variations, and then cut to length by a barrel-type flying shear. In addition, the coils are edge-trimmed as necessary on one of the lines.

Finally, the cut sheets pass through a classifier which separates them into three piles. Scrap that has been detected by a continuously operating thickness gauge or by the inspector, is placed automatically on the first pile; the second pile is made up of sheet of doubtful quality selected by the inspector; and prime quality sheets are collected in the third pile. The sheets classified as doubtful go for further examination to an inspection line.

#### Circle Line

Circles are blanked from strip up to 18 in. wide. After leaving the slitter the coils are unwound, flattened, and fed into presses which blank from 120 to 500 circles per minute, according to size. Emerging from the presses, circles that are off-gauge or faulty in appearance are diverted and the others are visually inspected on both sides before being automatically stacked and counted.

#### Packing and Dispatch

At the end of the finishing bays, the various products are collected, carefully packed to protect the high-grade finish, and then despatched.

## Aspects of Power Press Safety

THE need to focus the attention of employers and workers concerned in the use of power presses in industry on the importance of a new and energetic outlook on the guarding of these machines is emphasised in the latest edition of "Accidents—How They Happen and How to Prevent Them" \* which has just been published.

The booklet, which is based on certain accidents reported to the Factory Inspectorate, comments at length on accidents caused in the use of these machines, and describes in detail nine particular incidents. It notes the "very severe injuries" which have been inflicted on many thousands of workers, and adds that the problems posed in seeking an effective safeguard are not easy, especially in view of the nature of the tools on any given machine.

The ideal safeguard is described as one which permanently prevents all access to the danger area, but this ideal can only be achieved to a limited extent because of the practical needs of industry. Another factor is the unfortunately all too common failure to give consideration to safety when tools are designed.

Pointing out that the effective maintenance of all safety devices, especially guards of the movable type, is a highly important feature in securing safety for all workers, the booklet comments that in a number of factories there are still old type guards which have long ended their useful career and should be replaced.

Even if sound safeguards are provided, it continues, many accidents occur through failure in the factory organisation to ensure that guards are effectively in position before persons are set to work on the machines. The evidence of accident investigation shows that these faults might lie at the door of the management who fail to plan, or see that planning takes place to ensure that

safety has its part in the organisation of the factory.

At lower level there is sometimes a careless approach on the part of shop supervision which permits, sometimes by reason of urgency, that machines shall be used without the safeguards being effectively applied.

Tool setters have a special duty and responsibility in the prevention of power press accidents. But they were often tempted, quite wrongly, to suppose that while taking trial pressings there was no necessity to apply a guard. In consequence, many tool setters had suffered severe injuries.

Many workers have been injured because the tool designers have failed to give consideration to methods of feeding or removing the work without the use of the operator's fingers directly in the danger area. There is much ignorance among tool designers on these matters, and it is suggested that firms who have tool design departments should make special arrangements to ensure that this important point is brought to the attention of their design staffs.

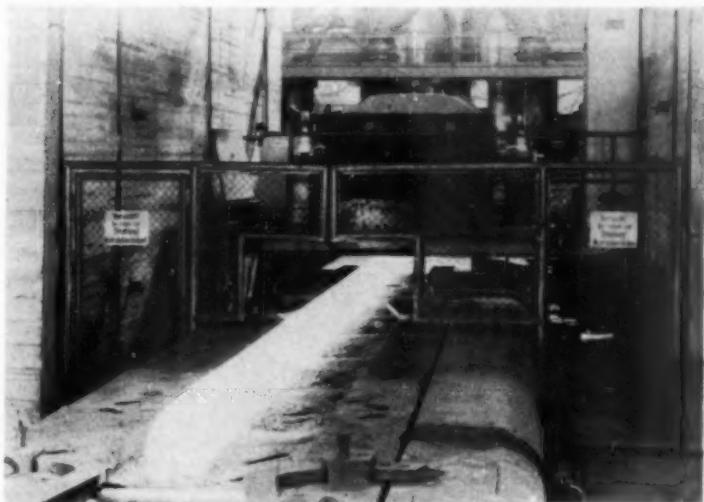
Admittedly, tool costs may be increased, but these are very frequently offset by the increased production, which may result from a well-designed tool giving scope for mechanical systems of feeding and removal. This subject is also linked with automation, which in itself can ultimately be an important contribution to accident prevention. This is achieved either by reducing the number of persons at risk, or by eliminating the risk through the provision of mechanical feeding and removal devices.

Of the annual toll of accidents at press tools, nearly one-half occurred at tools which were unguarded at the time. The reasons given for this were various, ranging from complete failure to see that a guard was provided, to neglect to use it when it was available and ready.

\* No. 45 H.M. Stationery Office, price 1s. 3d. net.

# X-Ray Inspection of Hot Steel Billets During Rolling\*

By W. J. Oosterkamp,  
J. Proper and  
M. C. Teves



*In an attempt to improve the finished product and to avoid needless waste of material by detecting cavities and inclusions during the rolling of hot billets, a trial installation was designed and constructed by Phoenix-Rheinrohr AG, Düsseldorf, in which the steel billets were moved through a beam of 31 MeV. X-rays generated in a betatron, and the image observed with the aid of a special X-ray image intensifier and an industrial television system. The results of screening several thousand hot billets showed that 50% of all holes 3 mm. thick were detected, and 90% of all holes 7 mm. thick.*

**I**N steelworks the molten steel from the open hearth furnaces, Bessemer converters or electric furnaces is cast into iron moulds, in which it solidifies into ingots weighing several tons. When solidification is complete, the ingots are removed from the moulds and transferred to the soaking pits prior to reducing in a cogging mill to billets or blooms some 30 ft. long and 8×8 in. or more in cross-section. Frequently the billets are immediately subjected to further rolling operations to reduce them to bars or sections only an inch or so thick.

A problem encountered in this procedure arises from cavities which may form in the ingot during solidification, which, if not welded up by rolling, materially weaken the finished product. Similar weakening may arise from slag inclusions in the steel. Experience has shown that defects of this kind are particularly prevalent in that part of the billet corresponding to the top end of the ingot, and it is therefore established practice to remove a length of several feet from this end and discard it as scrap. Inspection of the cut face for holes or inclusions reveals whether too much or too little has been discarded. The presence of such defects, however, cannot be very clearly observed on the red-hot metal. Consequently, it is quite possible that, in fact, too little will be removed, which means that poor-quality steel is

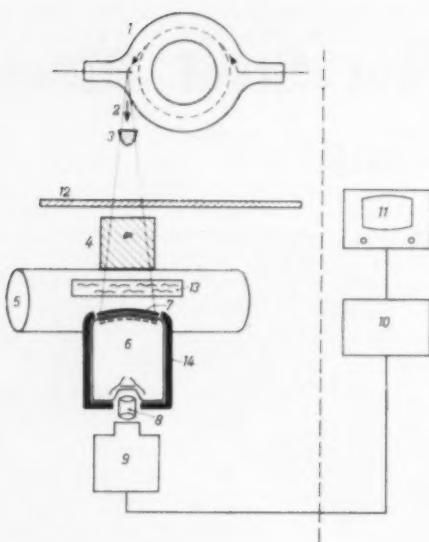
passed for further processing, or that, to be on the safe side, too much is cut off, which is a waste of good material.

It is evident that X-ray investigation of the hot billets in the rolling mill would result in a considerable saving of material, and also make for a more reliable product. Furthermore, it would show whether defects were present in parts of the billet other than the discard, and finally—and this is perhaps the most important point—it would provide a practical and simple means of investigating the effect of various production factors on the formation of cavities and other casting defects. It was indeed similar considerations that led to the widespread adoption of X-radiography for the inspection of welds; apart from its great usefulness for routine inspections, radiography has also contributed substantially to the improvement of welding techniques.<sup>1</sup>

In the present case it is a matter of detecting holes, or inclusions, measuring only a few millimetres across, in a steel billet at least 200 mm. (~8 in.) thick, and which, moreover, is moving at a steady rate of, say, a foot or two per second. For this purpose the normal X-ray equipment for industrial radiography, using X-radiation up to 300 keV., is hopelessly inadequate: the half-value

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<sup>1</sup> An alternative and widely used method of non-destructive testing is by means of ultrasonic vibrations; this is not suitable for examining hot billets, however, because of excessive damping in the material at the temperatures involved.



1. Betatron tube.
2. Beam of 31 MeV. X-rays.
3. Copper equalising absorber.
4. Steel billet moving along roller table under betatron.
5. Roller.
6. X-ray image intensifier tube.
7. Lead layer over aluminium window of 6.
8. Optical system.
9. Television camera tube.
10. Television video circuits.
11. Display tube.
12. Aluminium plate shielding the betatron from the heat-radiated by the hot steel.
13. Water-cooled heat-radiation shield for the image-intensifier tube.
14. Lead shield to prevent scattered X-rays from penetrating into the image intensifier.

Fig. 1.—Schematic cross-section of the trial installation at Phoenix-Rheinrohr, Düsseldorf, for visual X-ray inspection of hot steel billets during rolling.

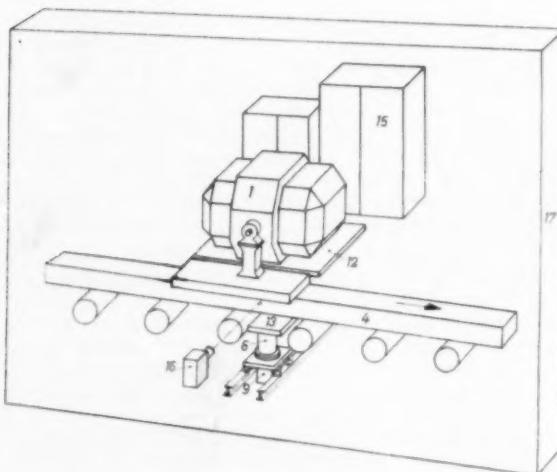
layer of steel for X-ray energies of 300 keV. is 6.5 mm. which means that this radiation is attenuated in steel 200 mm. thick by a factor of about  $2^{-30}$  or  $10^{-9}$ !

At the steelworks of Phoenix-Rheinrohr AG, Düsseldorf, plans were developed to tackle this problem with the aid of various modern devices: a betatron as radiation source, an X-ray image intensifier as detector, and an industrial closed-circuit television system for the final display. Preliminary tests and investigations, for which Philips made available a special X-ray image intensifier (designed for other purposes), led to the conclusion that the envisaged equipment would meet the requirements. One of the rolling plants at Phoenix-Rheinrohr was then equipped with a trial installation, and several thousand hot billets were subjected to X-ray fluoroscopy. Some particulars of this installation and of the results obtained are given below.<sup>2</sup>

The betatron radiation source, a product of Brown Boveri (Switzerland), generates X-ray energies up to

<sup>2</sup> For a detailed report see W. Lückerath, K. Fink and R. Flossmann, *Stahl und Eisen*, 1959, **79** (22), 1637.

31 MeV. This extremely hard radiation can be used for fluoroscopy of relatively very thick materials, especially since the betatron gives an appreciable intensity, comparable to that of an X-ray tube working on 300 kV., 10 mA. What is more, an X-ray energy in the region of 30 million electronvolts is about the most favourable that can be chosen in this case, the penetrating power in steel then being greatest; at higher energies the absorption in steel progressively increases as a result of pair production. Nevertheless, the half-value layer of steel for 31 MeV. radiation is only 34 mm., which is still much smaller than the object thickness. After traversing 200 mm. of steel, the rays are reduced in intensity to about  $2^{-6}$ , i.e. to roughly 1% of the initial value. Observation at this low residual intensity is made possible by the X-ray image intensifier, to which we shall refer presently. The third part of the installation is, as mentioned above, an industrial television system for remote display of the image formed on the viewing screen of the image-intensifier tube. This television equipment uses an image-orthicon camera tube and was

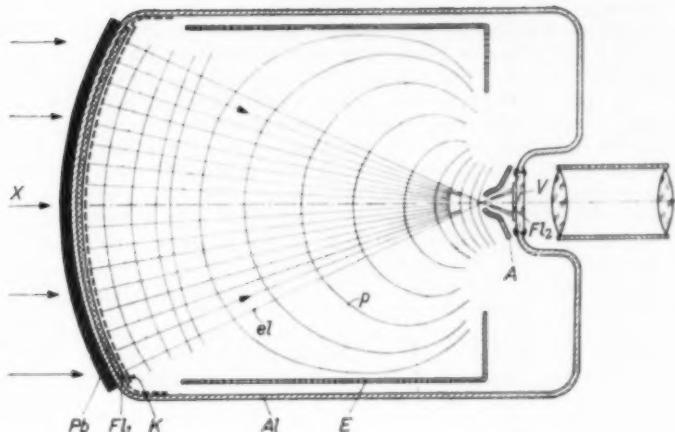


1. Betatron tube.
2. Steel billet.
3. X-ray image intensifier tube.
4. Television camera tube.
5. Aluminium plate shielding the betatron from the heat radiated from the hot steel.
6. Water-cooled heat-radiation shield for the image-intensifier tube.
7. Power pack for betatron.
8. Second television camera, enabling observer to keep an eye on the movement of the billet.
9. One of the concrete walls that help to shield the environment against X-radiation scattered from the billet.
10. Items 6 and 9 are mounted on a carriage to permit lateral displacement.

Fig. 2.—Perspective sketch of the installation.

Al Aluminium wall of tube.  
 Fl<sub>1</sub> X-ray screen.  
 K Photocathode.  
 E Focusing electrode.  
 A Anode.  
 Fl<sub>2</sub> Viewing screen.  
 V Glass viewing window.  
 el Electron path.  
 p Equipotential surface.  
 Pb Lead layer.

Fig. 3.—Cross-section of Philips 9 in. X-ray image intensifier placed at the disposal of Phoenix-Rheinrohr AG. A number of electron paths (el) and the cross-sections of a number of equipotential surfaces (p) are shown. For the purpose of the experiment a lead layer (Pb) was fitted over the aluminium wall covering the X-ray screen: the incident 31 MeV. X-radiation (X) releases electrons from this layer.



supplied by Fernseh G.m.b.H., of Darmstadt. A full-size image of the viewing screen is formed in the usual way on the photocathode of the image orthicon by a conventional optical system. The use of television offers several advantages. In the first place it is the easiest answer to the problem of safely positioning the observer, who cannot of course be immediately behind the intensifier viewing screen, where the level of residual radiation is still dangerously high. Further, the television system, with its extremely sensitive image orthicon, enables a luminance gain to be obtained that supplements that of the image intensifier. Finally, it is a simple matter in a television circuit to provide for contrast intensification.<sup>3</sup> All these favourable factors make it possible in the installation described to achieve better detail perceptibility than if the viewing screen were observed by means of mirrors and a telescope—a method which, although safe for the observer, is hardly a reasonable proposition in a steel-rolling plant.

Since a uniform brightness is obtained in the whole field of view during the screening of the rectangular-sectioned steel billets, a considerable degree of contrast intensification may be applied. To obtain a uniform intensity distribution across the X-ray beam from the betatron, a suitably shaped equalising absorber is placed in front of the betatron window.

The layout of the installation is illustrated in Figs. 1 and 2, and some additional details are mentioned in the captions.

The X-ray image intensifier works on the following principle: X-rays falling on a fluorescent screen excite an image, the photons of which release electrons from a photocathode in contact with this fluorescent screen. These electrons are accelerated by a high potential (25 kV.) and focused on a second fluorescent screen, the viewing screen. The image intensifier placed at the disposal of Phoenix-Rheinrohr (see Fig. 3) had a tube wall made entirely of aluminium and gave an electron-optical reduction of more than 10 times, and an overall luminance gain of about 1,200 times. The field of the

image intensifier, i.e. the diameter of the X-ray fluorescent screen, is 9 in.; this is the same as that of the recently introduced image intensifier for medical applications. It is thus an intermediate size between the types earlier described,<sup>4</sup> of 5 in. and 11 in. For use in conjunction with the betatron, a layer of lead 2 mm. thick was fitted over the aluminium window of the intensifier. This was done because only a very small fraction of the 31 MeV. radiation is directly absorbed by the X-ray screen and used for producing the fluorescent light. In the lead, the 31 MeV. X-rays release electrons that possess an energy of about 10 MeV. and travel in directions not very different from the beam of X-rays. These electrons pass through the aluminium window without appreciable scattering (the window is only 1 mm. thick) and in the fluorescent screen, fitted on the inside of the window, they excite about 4,000 photons of fluorescent light per electron. Image intensification then occurs as already outlined above, and each 10 MeV. electron from the lead gives rise finally to 400,000 light photons from the viewing screen.

It is worth pausing here to reflect on the remarkable sequence of particle interactions occurring in this equipment. *Electrons*, accelerated in the betatron, generate *X-ray photons* in the target, and these, after passing through the object, liberate in their turn *electrons* from the lead shield. These strike the X-ray screen, where they excite *light photons*, which again release *electrons* from the photocathode. These electrons, after acceleration, finally produce *light photons* again in the viewing screen. In the television system there is the further sequence of electronic phenomena by which the picture information is transmitted to the picture tube, where it is again converted to light photons that finally reach the eye of the observer.

The extremely high gain obtained in the X-ray image intensifier (and in the television circuit) does not alter the fact that the information, upon arrival in the image intensifier, is carried by a relatively low number of X-ray quanta. A high "noise" level in the picture is therefore inevitable.<sup>5</sup> The effect is reduced to some extent by the

<sup>3</sup> Contrast control, but usually with the object of attenuating instead of intensifying contrast, is common practice in television, a gamma corrector circuit being used for the purpose. See e.g. *Philips Tech. Rev.*, 1953/54, **15**, 227 et seq. The circuit there employed permits a contrast gain of up to 1.5×. Other circuits are used for obtaining the much higher contrast intensification required in the present case.

<sup>4</sup> Terres, M. C., and Tol, T., "Electronic intensification of fluoroscopic images," *Philips Tech. Rev.*, 1952/53, **14**, 33-43 (description of 5 in. tube). The 11 in. tube is described in *Philips Tech. Rev.*, 1958/59, **20** (11), 331-345.

<sup>5</sup> For a more detailed consideration of noise in X-ray images see: Tol, T., and Oosterkamp, W. J., "The application of the X-ray image intensifier, II. The perception of small object-detail," *Philips Tech. Rev.*, 1955/56, **17**, 71-77.

afterglow of the television picture tube, in which each image element is integrated over a certain time. Of course, the afterglow persistence may not be unduly long in view of the fact that the steel billet is a moving object.

In spite of the remaining noise, the trial installation gave surprisingly good results. Penetrometer measurements (i.e. determination of the thinnest wires still just perceptible) yielded the curve shown in Fig. 4. This indicates that, in a fluoroscopic examination of a steel billet 200 mm. (8 in.) thick, steel wires of 4 mm. diameter on the billet can still be observed. A statistical analysis was made of the casting defects found during the screening of several thousand hot steel billets at Phoenix-Rheinrohr, against the defects found during the subsequent inspection of cold sawn-off sections. The conclusion was that screening in this way discloses 50% of all cavities 3 mm. thick and 90% of all cavities 7 mm. thick.

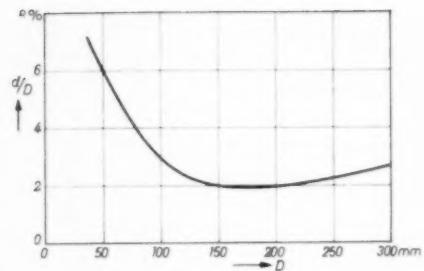


Fig. 4.—Perceptibility of thin wires on a steel billet (penetrometer test) during inspection with the installation described. The percentage thickness ( $d/D$ ) of the thinnest wire still just perceptible is plotted as a function of the thickness  $D$  of the steel.

## Automation in the Steel Industry

The first conference on automation in the iron and steel industry, organised by the British Iron and Steel Research Association, was held at the Palace Hotel, Buxton, on October 12th, with Mr. W. M. Larke in the chair. The opening address was delivered by Lord Halsbury, president of the Operational Research Society and one of the pioneers of computer technology in this country. He examined briefly the four types of operations which fell under the heading of the term "automation." Of these—transfer machining, assembling and packaging, batch and continuous process control, and data handling processes—the latter two, he believed, had the greatest applicability to the iron and steel industry. Lord Halsbury emphasised that the first step toward automation in any industry was the collection of operating data by means of full plant instrumentation. Only after complete operating data was available could the necessary operating rules be calculated for use with "on line" computers in the automatic control of processes.

Mr. S. S. Carlisle, an assistant director of B.I.S.R.A. with responsibility for automation matters, followed the opening address with an introductory review of automation in the industry. In this he discussed some sections of steelworks practice where the technology was already available to apply more automation. Examples were hot and cold strip mills, primary and plate mills and forging presses. There was, moreover, virtually complete instrumentation of the blast furnace and open hearth furnace. It was, however, still necessary in most instances, either to evaluate the case for further automation, or to learn yet more about the fundamental logic and technology of the processes, so that the rules that Lord Halsbury had spoken of could be determined and then built into the computer control.

A paper was then delivered by Mr. R. C. Gardner, of The Reed Paper Group on automation in the paper industry, and this was followed by a general discussion during which representatives from other industries contributed accounts of their experiences of automation development.

The final session of the conference was devoted to a brains trust, under the chairmanship of Lord Halsbury, consisting of : Mr. W. F. Cartwright, Dr. J. H. Chesters,

Mr. M. F. Dowding, Mr. R. H. Tizard, Mr. A. J. Young, and Mr. M. J. F. Coales. The intensive exchange of questions and answers that took place during this session was witness to the success of the conference, the primary aim of which was to stimulate the industry's interest in automation and suggest ways in which it might profitably be applied in the near future.

It was generally felt that this conference should be followed by others which would proceed to consider specific aspects of a subject of immediate technical and economic importance.

### Orders for Conveyors

WITHIN three months of the announcement of an agreement being signed between Brown, Lenox & Co., Ltd., of Pontypridd, and the Palmer-Bee Co. of Detroit, Michigan, U.S.A., orders have been booked for £400,000 worth of heavy duty conveyors and auxiliary equipment for steel rolling mills. These are to be built by Brown, Lenox & Co., Ltd., to the designs and specifications of the Palmer-Bee Co. The orders include coil conveyor equipment for the Spencer Works at Llanwern of Richard Thomas and Baldwins, Ltd., and for Colvilles new steel strip mill in Scotland. Another contract for similar equipment has been secured from the Steel Company of Wales, Ltd., for Velindre Works.

### Mineral Wool Production

PRODUCTION of mineral wool—an insulation material—has been improved and quickened by using oxygen at the Stillington, Co. Durham, works of Stillite Products, Ltd. Mineral wool is produced mainly from the oxides of silicon, aluminium, calcium and magnesium. The raw material is melted in coke fired cupolas. Melting takes place very quickly and, without oxygen, unevenly, because of the different constituents of the raw material. By boosting the temperature with an air-oxygen blast (oxygen provided by British Oxygen), melting is more even and there is a greater degree of control. There is also an appreciable speed-up in the actual melting process. Mineral wool is used extensively for heat and cold insulation, and as an insulant for liquid oxygen storage tanks. It is also used for sound insulation, acoustic correction, and as a filter medium.

# Semiconductors—Their Production and Properties

By W. H. Dennis, B.Sc., M.I.M.M.

*The properties peculiar to the class of elements known as semiconductors have led to a remarkable increase in use in electronic devices during the last decade. In this article the author refers briefly to the properties of semiconductors before proceeding to an account of the techniques used in the recovery and processing of the three principal ones—germanium, silicon and selenium.*

SINCE the advent of the transistor in 1948, increasing attention has been directed to a small number of elements, notably germanium and silicon, collectively known as the semiconductors, their electrical conductivity being intermediate between those of metals and insulators. So intensive has been the research and development work in this field that the semiconductors are seriously challenging the role of the thermionic valve which has for so long held sway in the rectification and amplification of electronic currents. The chief members of the group are shown in Table I, which includes for comparison a metal conductor and an insulator.

## Features of Semiconductors

The semiconductors exhibit a number of exceptional properties not found in any other class of materials, which are of great practical value and have been utilised in a number of ways. These properties can be briefly summarised as follows :

(1) The electrical conductivity of selenium is markedly affected by variation in the intensity of light, increase causing a decrease in resistivity. This property has been utilised in many directions, notably in the selenium photo-conductive cell. As an example, the cell has been adapted for automatically lighting and extinguishing the lamp in harbour buoys. As daylight fades the resistance of the cell increases, causing a switch to turn on the light. As night gives way to daybreak the reverse occurs, resistance decreasing, the light being switched off.

(2) The conductivity is likewise influenced by small changes in temperature, increase causing a decrease in resistivity.

(3) When a suitably prepared specimen is exposed to light, a flow of current is provided, the strength of which varies with the light source. With maximum illumination the voltage developed at present is about 0.3 volts : the assembly is known as the photo-voltaic cell. Probably the best known application of this principle is the photographic exposure meter, in which the cell is connected to a micro-ammeter calibrated in light intensity units. The latest use of these cells is in artificial satellites and other space vehicles, in which the solar-powered cells provide its radio-transmitter with sufficient current to keep contact with stations on the earth.

(4) The conductivity and other electrical properties of germanium and silicon are greatly influenced by the presence of small amounts of other elements, and thus these properties may be altered or varied at will by the regulated addition of impurities.

TABLE I.—ELECTRICAL RESISTIVITIES OF SEMICONDUCTORS  
(microhms per centimetre cube)

| Material  | Type          | Resistivity     |
|-----------|---------------|-----------------|
| Copper    | Conductor     | 1.56            |
| Germanium | Semiconductor | $10^8$          |
| Silicon   | Semiconductor | $10^8$ — $10^9$ |
| Selenium  | Semiconductor | $8 \times 10^8$ |
| Glass     | Insulator     | $10^{14}$       |

(5) When used in conjunction with suitable contacts, current from an external source flows much more readily in one direction than the other. In effect, the current becomes uni-directional, alternating current being rectified to direct. Whilst this characteristic has been utilised in the selenium rectifier for many years, germanium and silicon diode rectifiers are of more recent application, but are rapidly establishing themselves because of small size and superior rectification and amplifying efficiency. Recent typical applications to the metallurgical industry include electrolysis, sodium production and copper refining.

(6) The discovery of the amplifying properties of a germanium crystal triode was made in 1948 by the Bell Telephone Company in the U.S.A. This was named a transistor and consisted essentially of two phosphor bronze wires making contact with the surface of a germanium crystal. This point contact type has now been largely replaced by the junction transistor, consisting of germanium or silicon crystals of two types induced by the addition of regulated amounts of other elements.

(7) In a somewhat different field, semiconductor refrigerators have been developed capable of achieving temperatures in the region of minus 40° C. and which use neither chemicals nor electric motors. The operation of these new machines depends on the use of very small compact units of semiconductor material which provide a strong cooling effect when an electric current is fed in. Such refrigerators are inherently highly efficient, they are compact, they require no moving parts, and they do away with the necessity of a cooling liquid.

The advantages of the transistor compared with the thermionic valve are that no current is required for heating and no vacuum is necessary. Moreover their small size and robust construction have certain obvious technical and economic benefits.

The disadvantages are that in addition to high cost (pure silicon costs £100 per pound), the amount of power handled at present is limited to a few watts, and

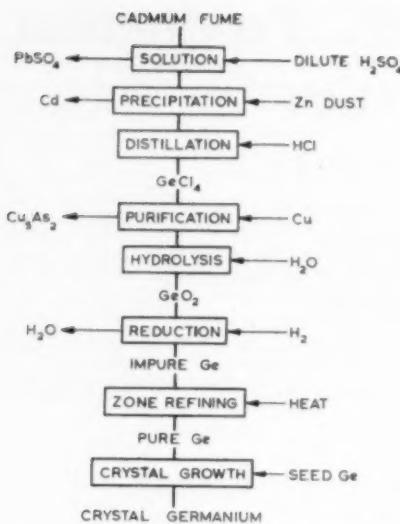


Fig. 1.—Flowsheet for germanium production.

also the ability to amplify over the whole range of frequencies used in radio and industrial electronics is limited.

However, the advantages of both the semiconductor diodes and triodes over the more conventional devices are such that many millions are now being produced every year, and it would seem certain that their scope will be considerably extended as research succeeds in bringing down the cost and overcoming their power limitations.

#### Recovery and Processing

The production of germanium, silicon and selenium for electronic usage require that they be processed to a high degree of purity, for the properties of the material are very sensitive to the influence of foreign atoms in their structure. Having secured the element in a pure form, further processing is necessary in the case of germanium and silicon to promote the production of single crystals necessary to secure the requisite properties. As the raw material and methods used in the preparation of the materials differ in considerable detail it is necessary to consider each element separately.

#### Germanium

Though of widespread occurrence in the earth's crust, germanium is not found in quantities large enough to permit of economic extraction, output resulting solely as a by-product as a consequence of the treatment of other materials. The chief commercial source is the cadmium fume resulting from the processing of zinc ores. This forms the raw material for the largest producers, namely America and Belgium. Germanium is also present in certain types of coal and this forms the chief British source, the material concentrating in the flue dusts from boiler installations, gas producer plants, etc., containing up to 0.75% germanium. Production of germanium in this country was initiated some eight years ago by Johnson Matthey & Co., Ltd., the precious metal refiners. From whatever source, the element is separated (Fig. 1) from contaminating materials by distillation of its tetrachloride from strongly hydrochloric

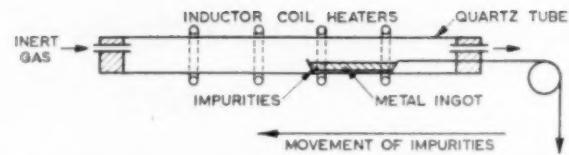


Fig. 2.—Diagrammatic representation of the method employed in zone refining.

acid solutions. The chief impurity—arsenic—also forms a chloride, and it is almost impossible to separate the two chlorides by fractional distillation, since arsenic tetrachloride is appreciably volatile at the distillation temperature of germanium tetrachloride. The method of separation in general use involves the use of copper, with which arsenic tetrachloride reacts to form cuprous chloride and copper arsenide, whereas germanium tetrachloride is unaffected. Refluxing of the chlorides in the presence of copper thus serves to eliminate the arsenic. Hydrolysis of the tetrachloride with water results in the precipitation of germanium dioxide, which is filtered off, washed and dried. Reduction of dioxide to metal is carried out by heating in a current of hydrogen at a temperature of 600–650°C., a final temperature of 1,000°C., consolidating the metal powder into ingot form.

The metal still contains trace amounts of such impurities as arsenic, antimony, indium, sulphur and iron, and further purification is thus necessary. This is generally achieved by a process known as zone refining and is based on the segregation of impurities between a molten and a solid part of the material under the influence of heat. An ingot of the metal contained in a quartz boat is placed in a long quartz tube through which circulates an inert gas such as argon. Heating is secured by surrounding the tube at intervals by induction coils which are so arranged as to travel slowly along the axis from one end of the ingot to the other, thus enabling several molten zones to traverse the ingot at the same time. The impurities present tend to remain in the molten zone and concentrate in that end of the ingot towards which the coils are moving leaving the rest of the ingot in a pure condition. By breaking off the impure end of the ingot and repeating the process, purification to a high degree may be effected. In some forms of zone refining the coils are stationary, the crucible containing the metal being caused to travel slowly through the length of the quartz tube (Fig. 2).

#### Formation of Single Crystals

This is achieved by controlled solidification in the presence of a specimen specially prepared from a previous batch and known as the seed. The zone-purified

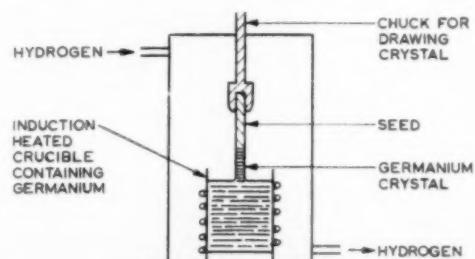


Fig. 3.—Sketch of apparatus for crystal drawing.

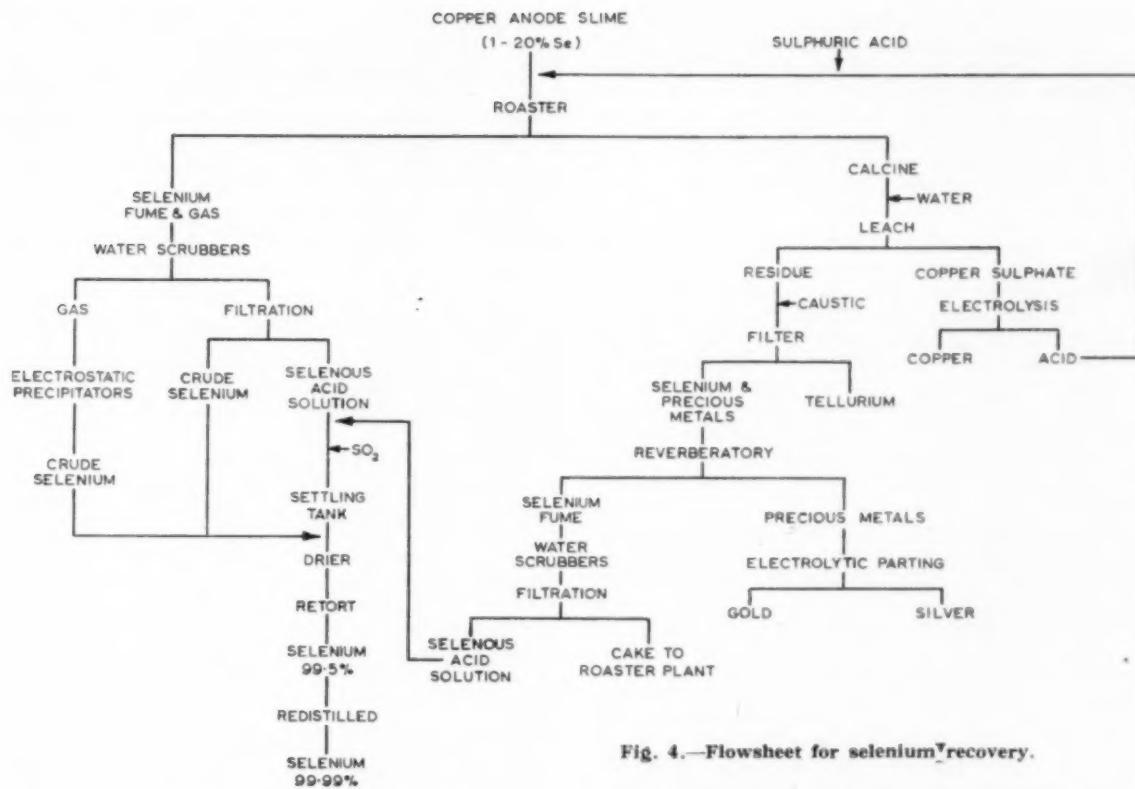


Fig. 4.—Flowsheet for selenium recovery.

germanium is melted in a crucible (Fig. 3) and the seed in rod form is then lowered on to the surface of the molten metal. Carefully controlled conditions keep the temperature high enough to melt the tip of the seed and low enough for a meniscus of liquid metal to be held up from the molten metal by surface tension. When a sufficient diameter of solidification has been formed around the tip, the seed is automatically raised at about a rate of three inches per hour. This results in a growing column of solid germanium being pulled up from the melt in the form of a single large crystal. The operation is carried out in an atmosphere of argon or hydrogen to obviate any atmospheric contamination. The introduction of other elements to attain some desired attribute is done by adding to the crucible a weighed amount of the requisite material.

The single crystal is shaped into a square section and cut into 0.02 in. wafers by means of a small precision saw of a type used for cutting diamonds. Finally, the wafers are cleaned and given the necessary surface finish by etching in acid. Recently a method of growing germanium crystals as thin uniform flat ribbons has been developed. This eliminates the slicing into thin wafers, grinding, cutting into small squares, and finally polishing.

#### Silicon

Next to oxygen, silicon is the most plentiful element in nature, for it is the main constituent in sand, soil and rocks. It is never found uncombined, but nearly always linked with oxygen as the oxide, silica. In Great Britain the chief producer is Imperial Chemical Industries, Ltd., with an output of approximately 2,000 lb. per annum.

The raw material used in the production of silicon is quartz sand or sand stone which has been crushed and ground, washed free of clay and loam, and acid leached to remove iron oxide. Reduction of the oxide to metal is carried out in an electric arc furnace in the presence of carbon. Metal of 98-99% purity is produced. Substantially pure metal is obtained by fractional distillation of the tetrachloride followed by reduction with zinc, the chloride, together with zinc vapour, being led through a quartz tube held at a temperature of 1,000° C., pure silicon being deposited on the walls of the reaction vessel. Alternatively, the tetrachloride can be reduced by lithium aluminium hydride to yield gaseous silane ( $SiH_4$ ). By liquefaction and fractionation a substantially pure product can be obtained which, on decomposition at temperatures above 400° C., breaks down into silicon and hydrogen. The principal trace impurities in the silicon are generally boron and phosphorus—of the order of a few hundredth parts per million—at which level the silicon is sufficiently pure to render unnecessary any further refining treatment. Zone refining is, however, practised where necessary. The technique of producing single crystals by vertical pulling is essentially the same as that for germanium.

#### Selenium

Selenium, true to its chemical similarity with sulphur, forms compounds analogous to sulphides as in the selenide minerals naumanite ( $Ag_2Se$ ), clausthalite ( $PbSe$ ) and eurairite ( $AgCu_2Se$ ). Sulphides and selenides frequently occur together, practically the whole output of selenium being derived from the treatment of Canadian

and American copper sulphide ores. When these ores are smelted, the selenium accompanies the copper through the successive reverberatory and conversion treatments, being concentrated in the blister copper which may contain up to 0.1% selenium. During the subsequent electrolytic refining, the selenium, being insoluble, remains either on the anode or gravitates to the bottom of the tank as slime with other insolubles.

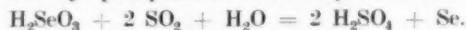
The selenium content varies from 1 to 20%, with 10-40% copper. Since the slimes contain tellurium, gold and silver, their separation and recovery is an integral part of recovery operations (Fig. 4). The slime is roasted with 50% of its weight of sulphuric acid, the copper content thereby being rendered water soluble. Selenium, being highly volatile, vaporises during the operation and is exhausted to water scrubbers and electrostatic precipitators. Some 75% of the selenium is eliminated during the roasting treatment, being recovered as crude metallic selenium and selenous acid solution.

Water leaching of the roasted slimes removes the copper as copper sulphate, the copper being recovered by electrolysis, the sulphuric acid liberated during the process being concentrated for re-use in roasting. The leached slime is treated with 10% caustic soda which dissolves most of the tellurium, its recovery being effected by neutralisation with acid, tellurium precipitating as the dioxide. The caustic-leached slime containing gold, silver and the remainder of the selenium is charged to a small reverberatory together with soda ash and silica. The slime readily melts and forms a slag which is skimmed off. This slag contains most of the minor impurities such as lead, arsenic and iron. Nitre and more soda are added and a further slag containing residual copper is removed, the furnace metal

then being cast into anodes, from which the gold and silver is recovered by electrolysis.

The gases leaving the reverberatory furnace containing most of the residual selenium are scrubbed, the resulting sludge being filtered, the solution pumped to the selenium recovery plant, and the filter cake containing silica, arsenic, selenium, tellurium, etc., returned to the roaster for retreatment.

Recovery of selenium from the selenous solution is effected by precipitation with sulphur dioxide gas



The precipitated selenium is allowed to settle, the supernatant solution being decanted and pumped to the acid recovery plant. The settled selenium is filtered, dried and refined by distillation in iron retorts. Retorted metal assays 99.5% selenium, and for electronic usage is redistilled to yield a 99.99% purity product.

The world output of selenium—500 tons per annum—is on a considerably greater scale than either germanium (45 tons per annum) or silicon, for in addition to its use as a semiconductor it has many other outlets, notably in the glass industry. Small amounts (0.01%) added to the ordinary grades of glass neutralise the undesirable green tint due to iron, yielding a crystal clear product in demand as bottles and containers by the food and drink, cosmetic and pharmaceutical industries. Much larger amounts (2.5%) are incorporated for the production of ruby red glass for car rear lights and railway signal lamps.

The characteristic of selenium of imparting a red colour has also been utilised in the pigment and paint industries. Combined with cadmium, such materials are also used in the plastic and rubber industries for producing reds superior in non-fading and heat-resisting properties as compared to organic pigments.

### Fatigue Testing Co-operation

STEEL subjected for a short period to extreme stresses reacts in a manner diametrically opposed to that of some light alloys. This discovery was announced by Dr. Hempel of Germany at a meeting of the Working Party on Fatigue Failure in Metals, organised by the European Productivity Agency of the O.E.E.C. This working party was set up because a series of failures—among them being the accidents to certain airliners—revealed that too little was known about the nature and causes of fatigue in metals. It was decided that at least five thousand testpieces should be tested to destruction, particular emphasis being laid on the effects of variations in the degree of stress applied.

To attempt to carry out this work in a single laboratory, or even a single country, would take far too long, and this is one of the instances where international co-operation, co-ordinated by a central organisation, can be so useful. In fact, the tests are being carried out in nine European countries, in the United States, and in Canada. So far some five hundred testpieces have been used, chiefly with the idea of establishing whether the working methods and the stresses agreed on in advance were suitable. They have proved to be so, and work is now about to begin on the main batch of five thousand testpieces. They will be tested for varying periods at four different stresses, known for convenience

as S1, S2, S3 and S4. The value of S4 used by Dr. Hempel was 70 kg./sq. mm.

Although these preliminary tests were not expected to yield any results, it rapidly became apparent that steel subjected to stress of this severity for even a short time subsequently cracked up very fast under much lower stresses—S1 or S2. The effect on various light alloys, however, was to harden and strengthen them so that they subsequently stood up better to prolonged periods of moderate stress than they did when not subject to an initial period of high stress.

As M. Crussard, of the Institut de Recherche de la Sidérurgie, Chairman of the Working Party, pointed out, it is extremely heartening to have even a partial result at this early stage, and one which obviously gives a useful lead for further experiments. The main body of tests, spread out over eleven countries, will be completed within a year, and the results will go a long way towards making machines safer and more durable.

SCHLOEMANN AKTIENGESELLSCHAFT, Düsseldorf, are building a continuous wire rod mill for the Klöckner-Werke A.G., Hütte Haspe, which will comprise a roughing train consisting of eight stands, two intermediate trains each having four stands and a finishing train with eight stands.

# The Advantages of Aluminium Foil for Transformer Windings

By L. W. Swann

(*Fisher's Foils, Ltd.*)

THE use of aluminium as a conductor material as an alternative to copper is well-established in the electrical engineering field, although there is not at the moment any signs of the one metal completely displacing the other. To date the main applications have involved wire or extruded sections, although foil has long been used in the production of capacitors. During recent years, however, attention has been directed to the replacement of copper wire by aluminium foil for the windings of transformers.

As copper is in wire form and as aluminium for this application is in strip or foil form, the same coil-winding machinery cannot be used, and the prospect of having to change some of their plant may have deterred transformer manufacturers from giving to aluminium the attention that its advantages, both technical and economic, warrant. Aluminium foil is particularly suitable for the windings of small instrumentation transformers.

## Aluminium Foil v. Copper Wire

Annealed aluminium has an electrical resistivity of 2.665 microhm cm. against a figure for copper of 1.588. Thus, to carry a given amount of current, the volume of aluminium must be some 60% greater than that of copper. This does not, however, mean that the resultant aluminium foil winding would necessarily be larger than one made conventionally from copper wire. In any winding consisting of round wire there must be considerable air space, for in its cross-section one wire is in contact with those around it only at points. The use of flat aluminium foil enables one turn to be in complete contact with the next, thereby permitting the total area occupied by the winding to be reduced to a minimum.

Copper is nearly three and a half times as heavy as aluminium. Whilst as shown above, one needs 60% more aluminium than copper by volume to achieve the same electrical conduction, the greater relative weight of copper causes an aluminium foil winding to weigh only about half that of a copper wire winding of equivalent performance. As so much electrical apparatus nowadays has to be portable or designed for use in vehicles, and especially in aeroplanes, lightness is of considerable importance.

Although machines for winding copper wire transformer coils cannot be used for producing aluminium foil coils, machines for the latter can be much simpler in construction, for there is no need of a traversing mechanism such as is required to wind wire in layers. A winding of aluminium foil can also be made much more quickly. The costs, therefore, of making the necessary change in production techniques would not be so high as might at one time have been feared; in any case,

they would be followed by immediate economies both in labour and in the purchase of the raw materials.

## Insulation of Windings

In insulating one turn from the next in an aluminium foil transformer coil one has the choice of several methods. Firstly, the foil may be wound up with paper or film such as Melinex. It is, however, recommended that the completed foil be sealed or impregnated by one of the varnishes so widely used in the electrical trade. By this means, increased rigidity is given to the coil, whilst, at the same time, guarding against the penetration of moisture. Most varnishes are designed to withstand temperatures up to 90° C., although, in cases where the heat generated by the transformer is greater, silicone and epoxy resins should be employed, which are satisfactory up to about 200° C.

A much-favoured method of providing insulation for aluminium foil windings is that of anodising. It has two great advantages. Firstly, the anodic coating will withstand high temperatures; secondly, by being so very thin—seldom exceeding 0.0002 in., and thus much thinner than interwound insulation such as paper or film—it enables the size of the finished coil to be reduced to an absolute minimum. A reason that this technique has not hitherto been adopted to an extent warranted by its merits is the limited facilities available in Britain for continuous anodising. During the next year or so there is likely to be more of such plant brought into operation. This in itself should do much to promote further the manufacture of aluminium foil transformers.

The third means of providing inter-turn insulation—still under investigation—is one which, when brought finally to perfection, will possess many advantages. The method entails the application to the foil, by means of a suitable coating apparatus of a resin or lacquer of the two-stage type, which is then dried off sufficiently to enable the foil to be reeled up. The foil is then slit into reels of suitable width, which are used by the transformer manufacturer for winding the coils. These are afterwards submitted to a heating, or final curing, process, when the coating softens and fuses. On cooling, the coil becomes one solid piece.

The merits of this technique are that by a simple coating applied to the foil one provides the inter-turn insulation and essential rigidity. The thermo-setting property of the coating is designed to ensure that it will withstand the heat that might later be generated within the coil during use. Although there are certain problems yet to be overcome before this method of manufacture can be adopted, there is every reason to suppose that it will revolutionise the construction of the instrumentation type of transformer.

## Electrical Conduction Equivalents

In an admirable paper by Rata and Haggitt, of Alcan Industries, Ltd., which appeared in the June, 1960, issue of *Electrical Times*, the authors give many detailed technical recommendations of the utmost value to transformer manufacturers wishing to undertake development work upon foil windings. Of particular interest is Table I, which provides a selection of foil sizes representing the equivalents in electrical conductivity of round copper wires in several different gauges.

In arriving at an aluminium foil equivalent of any round copper wire, one should take the cross-sectional area of the wire and increase it by about 64% to allow for the higher resistivity of the aluminium. The new cross-sectional area for the aluminium thus arrived at may then be expressed in width and thickness of many combinations, as illustrated in the Table. For example, a narrower coil may be made with thicker foil; whilst a coil whose overall diameter must be limited can be made with thinner but wider foil.

These variations are easily accomplished by a direct winding-up of the selected foil—a process far simpler and more rapid than the winding of copper wire. Moreover, foil windings do not demand the more expensive flanged bobbins needed for coils wound from wire; simple tubular centres are all that are needed. It is evident that, once the initial manufacturing arrangements have been made for winding aluminium foil coils, there is scope for an appreciable reduction in the production cost of transformers.

Whilst some emphasis has been placed above upon certain equivalent aspects of copper wire and aluminium foil in transformer construction, there is little doubt that a foil winding offers several technical advantages. Amongst these is a quicker dissipation of the heat generated during use, as a result of the elimination of the innumerable air spaces within a winding composed of round copper wire which constitute a barrier to the passage of heat.

## Jointing Techniques

A manufacturer contemplating the production of aluminium foil transformers will need to satisfy himself concerning the best way of making the necessary electrical connection with the foil winding. There are a number of recommended techniques, and the choice would probably be governed by the thickness of the foil constituting the winding, whether or not intermediate tappings had to be taken, and to what the foil had to be joined—whether another aluminium surface, or, for example, a copper wire.

Much research has already been undertaken upon the methods of making electrical joins with aluminium foil. One of the most effective is undoubtedly cold-pressure welding, necessitating only a simple hand tool, which, in bulk production, could be made to operate mechanically. When sufficient pressure is exerted by means of appropriately shaped dies, the metals forming the surfaces to be joined will flow, forming a true weld with a continuous grain structure. An advantage of this technique is that no heat is required and that aluminium foil may be joined with copper.

The established means of joining aluminium by tungsten arc welding and by soldering are not really suitable for metal in foil gauges of 0.006 in. (0.15 mm.) downwards. The most promising technique for making

TABLE I.—EQUIVALENTS IN ELECTRICAL CONDUCTIVITY

| Copper Wire<br>(s.w.g.) | Aluminium Foil<br>(width and thickness—in.) |
|-------------------------|---|
| 2                       | 20 x 0.005                                  |
| 6                       | 15 x 0.0032<br>10 x 0.0048                  |
| 10                      | 10 x 0.002<br>5 x 0.004                     |
| 14                      | 8 x 0.001<br>2 x 0.004                      |
| 18                      | 2 x 0.0015<br>1 x 0.003<br>½ x 0.006        |
| 22                      | 2 x 0.0005<br>1 x 0.001<br>½ x 0.002        |

connections, especially with the thinner foil, is that of ultrasonic welding. In Britain, as far as it is at present known, this method has not been appreciably developed beyond the laboratory stage. In America, on the other hand, the Sonobond Corporation now offers equipment for joining foil to copper tabs or wire for electrical application in the manufacture of transformers.

The plant for this purpose will make joins by either single spots, overlapping spots to produce a continuous bond, or a continuous weld using a rotating welding tip. For joining foil and copper wire a small bench type 300 W. ultrasonic unit is available. It is rapid in action, each weld taking between half and one and a half seconds. As there is no arc or spark, and as no electrical current flows through the surfaces to be joined, the process is suitable for women operatives. Surface preparation is unnecessary beyond degreasing. There is practically no deformation of the metal, thereby making the technique very suitable for foil joining. Most vibratory welding has been undertaken hitherto with a frequency range of 4,000 to 40,000 cycles per second. It would appear that metal of foil gauges responds best to the higher frequencies.

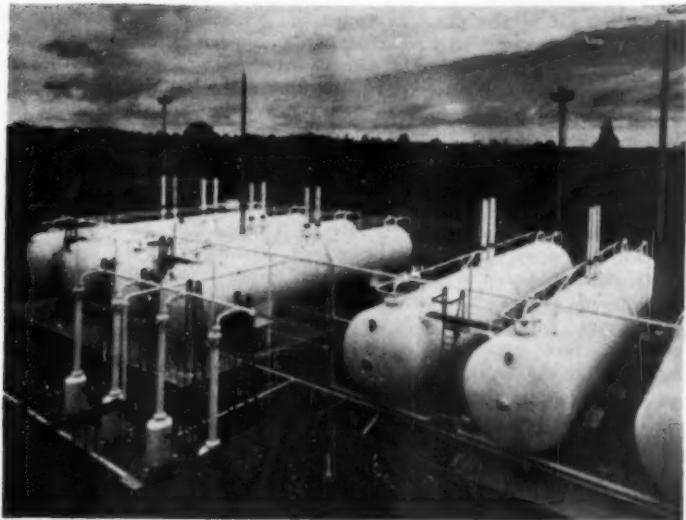
## Market Prospects

From all of the above it will be seen that the most thorough research has been carried out upon all major aspects of constructing the windings of instrumentation transformers from aluminium foil. The slowness of manufacturers to adopt the new technique is to be attributed to the long standing of the present copper wire usage; the fact that this is affording no appreciable dissatisfaction; a natural reluctance to relinquish existing plant for alternative plant of different—even if simpler—design; and the fact that the price of copper itself has descended somewhat from the elevated levels it reached during the American stock-piling programme.

Technical progress, not only in Europe, but throughout the world will, as time goes on, call for ever-increasing quantities of instrumentation transformers and other electrical components needing light-weight windings. The aim of manufacturers could well be initially, not the scrapping of their present copper wire winding equipment and replacing it with plant designed for use with aluminium foil, but, as demands grow, augmenting their existing equipment with the new. In other words, to undertake this important development within the movement of general expansion. Although a number of hitherto backward countries are in various stages of unrest, this unrest must be regarded as the inevitable prelude to material advancement. As the years pass they will become markets for British goods of ever-increasing importance.

# Liquefied Petroleum Gas Plant

## Hereford Installation the Largest in the Country



View of the plant showing some of the storage vessels with the four vaporisers in the foreground.

ALTHOUGH W. C. Holmes & Co., Ltd., of Huddersfield, have designed and installed numerous liquefied petroleum gas plants during recent years, the recently commissioned butane/air plant at the Hereford works of Henry Wiggin & Co., Ltd., is of particular interest, as it is the largest and most comprehensive installation in this country outside the gas industry. The plant has been designed to store 500 tons of liquid butane (the equivalent of some 240,000 therms), and to supply the total gas requirements of a modern factory of fifty acres.

In operation, butane vapour is mixed with air. This serves a dual purpose: it reduces the high heat content of the vapour to a usable value, and it also reduces its dew point (the temperature at which vapour condensation takes place). This reduction of dew point eliminates the possibility of the vapour separating from the air and condensing in the distribution pipe-lines. The butane/air mixture may be used in place of coal gas, providing suitable burner adjustments are made where necessary.

The liquid butane is transported from the refinery to Hereford in specially designed road tankers. To speed up "turn-round" of the tankers, a special method of liquid transfer, known as the vapour pressure method, has been adopted. Compressors draw vapour from the storage vessels, compress it and pass it into the tanker. This displaces the liquid which flows from the tanker into the selected storage vessel. When the liquid ceases to flow (both visible and audible warning is given of this event) the valve system of the compressor is reversed so that the vapour remaining in the tanker is transferred to the storage vessel. The advantages of this system are (a) rapid discharge of the liquid butane, and (b) the tanker is emptied not only of liquid, but also of virtually all the vapour.

There are eight storage vessels at Hereford, each of which is 10 ft. diameter  $\times$  56 ft. 6 in. long. When these vessels are filled to 86% of their total capacity the required total of 500 tons of liquid butane is reached. The remaining 14% of capacity forms the vapour space

and allows for expansion of the liquid. Each vessel is fitted with multiple safety relief valves which are fitted on special manifolds to facilitate the removal of individual valves for inspection purposes. The number of valves is such that at all times the vessel is fully protected. A float-operated liquid contents indicator shows the contents of each vessel, and remote reading repeaters are provided on the central control panel. Excess flow limitation valves, designed to close in the event of a sudden excess vapour flow, are fitted in the vapour off-takes. Special valves are also fitted on the liquid off-takes. These latter valves are maintained in the appropriate position by means of hydraulic pressure which is automatically released should the pipe-lines suffer mechanical damage.

Each vessel is fitted with water spray manifolds which, when necessary, can supply an adequate covering of water. This acts as a protection from either an abnormally high ambient temperature or radiated heat. As water supplies were limited on site, a small reservoir has been incorporated in the plant, the return water being drained from underneath the vessels and recirculated by means of submersible water pumps.

There are four vaporisers, each of which consists of a vertical cylindrical pressure vessel containing internal heating coils which are heated by saturated steam. Each vaporiser has been designed to produce 24,000 cu. ft./hr. of butane vapour, and as on the storage vessels, excess flow valves, safety relief valves and pressure gauges are fitted. After vaporisation, the butane vapour is passed through suitable stages of pressure reduction to the inlets of six Holmes-Kenip carburettors where the butane, at near atmospheric pressure, is mixed with the correct amount of air. Since the butane/air mixture is required at a pre-set and constant calorific value, the ratio of butane to air must be closely governed. The ratio is set manually, each carburettor being tuned to the Wobbe index recorder. When the correct setting is obtained the hand wheel is locked and the ratio of butane to air remains constant over the whole range of output.

The output of the plant is under the control of the plant operator, who ensures that the potential of the carburettors in operation is always higher than the factory demand as registered by the flow meter on the central control panel. Minute to minute variations of demand are absorbed by the automatic features of the carburettor units. The butane/air mixture is passed into an 18 in. diameter main which feeds the factory distribution network at approximately 2 lb./sq. in. As the plant is required to operate 24 hours a day, all electrical controls and gauges have been positioned on a central control panel to reduce personnel requirements on the various shifts. The panel houses: pressure gauges for steam, butane and butane/air; tank contents gauges; flow and Wobbe number recorders; and indicator lights to indicate high and low Wobbe, low

butane and butane/air pressures. The panel also incorporates stop and start buttons for the six mixing unit motors and for the air supply fan motor.

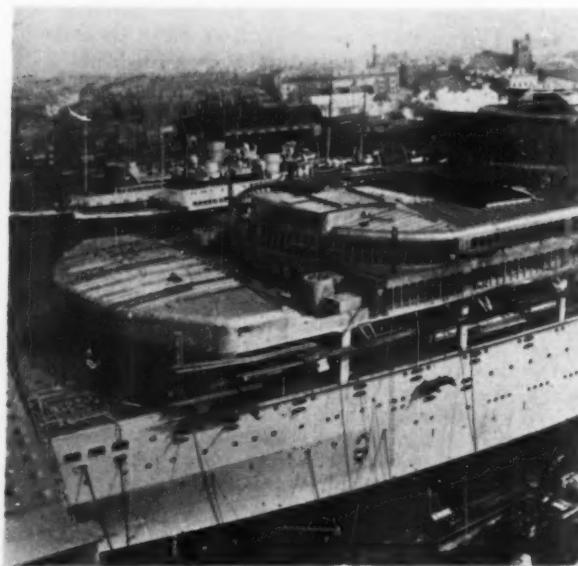
A system of safety devices and interlocks is provided on all mixing units so that, in the event of incorrect operation, or failure of butane or electrical supplies, alarms first indicate the fault and then, if the fault is not corrected, the plant automatically shuts down. In designing this plant, particular attention had to be given to the safety precautions peculiar to liquefied petroleum gases, and steps have been taken to meet all foreseeable eventualities. The absence of specific legislation covering the installation of butane/air plants necessitated the close co-operation of the local authority and appropriate government departments: every help was given by all concerned, particularly the Factory Inspectorate.

## Aluminium in the "Oriana"

THE *Oriana*, due to sail on her maiden voyage this month, is not only the largest passenger liner completed in a British shipyard since the war, but is also unique in embodying the largest welded aluminium structure yet made. Aluminium has been used in the *Oriana* for the whole of the structure above the strength deck, comprising in all eight tiers of houses, three of which are complete decks extending the full 450 ft. length of the superstructure. The total quantity of aluminium used is about 1,000 tons, of which 900 tons were supplied by Alcan Industries, Ltd. (formerly Northern Aluminium Co., Ltd.). In order to supply the largest possible plate sizes, the greater part of this material was produced in the form of composite welded plate (Noral B54S, B.S.1477:NP 5/6) made at Alcan's Rogerstone Works by the automatic welding and subsequent stretching of two pieces of smaller plate, a process that has, however, since been rendered unnecessary by the completion of a 144-in. plate mill at the same works. After welding and

stretching, all plates for the *Oriana* were sawn to the required dimensions and supplied to the shipyard with guaranteed parallel edges and a close tolerance on width in order to minimise the amount of subsequent fabrication.

The general principle followed in the welding of the aluminium superstructure has been to align plate edges with stiffeners wherever possible, the stiffeners thus providing permanent backing bars for the butt welds. This method of construction has been successfully applied in previous ships. Its use in the present instance was decided after a large scale trial carried out jointly by Alcan Industries and Vickers-Armstrongs some time before building of the ship began. For stiffening members generally, a range of tapered-bulb sections was used in preference to the better-known square-bulb type of aluminium section, and special extrusions were also used wherever possible for girders and other members that in a steel structure would normally be fabricated from plate. These extrusions (Noral alloy A56S, B.S.1476:NE6) ranged in size up to 16 in. overall dimension, with corresponding weights of up to 15 lb./ft.



### Electro-Erosion Machines

WICKMAN, LTD., under an agreement recently concluded with Ateliers des Charmilles, S.A., Geneva, have assumed the sole selling agency in the United Kingdom for the Eleroda Model D.1. electro-erosion machine. The Eleroda machine is already well known in this country as a high-precision spark machining unit particularly designed for tool-room applications, and its addition to the Wickman range will complement the present range of Erodomatics manufactured by the company. These include the Type W/BM bench or cabinet mounted machine incorporating vernier positioning to the electrode head and designed for spark-machining high precision components of small size; the Type W/SM self-contained medium size machine with simplified controls, specifically designed for production use; the recently introduced Type W/DM large capacity die machine available with 4.5 kW. or 13 kW. power pack and designed for the machining of heavy forging dies. The range is handled by the Wickman Electro-Mechanical Department, Upper York Street, Coventry.

# New Theory for Rusting of Iron

## Role of Hydrogen Ions in Promotion

**S**CIENTISTS at the Westinghouse Research Laboratories have advanced a new basic explanation for the rusting of iron—one of the most destructive and wasteful chemical reactions known to man. The basic cause of iron corrosion has been largely a scientific mystery. The new theory, proposed by the Westinghouse research workers—Dr. E. A. Gulbransen and Mr. T. P. Copan—suggests for the first time that the true cause is hydrogen ions, or protons. The hydrogen ions come from water vapour, which must be present if iron is to rust to any great extent at room temperature. The new theory suggests that the tiny hydrogen particles penetrate the iron and enlarge the sites at which oxygen normally combines with the metal. This spreads the reaction throughout the surface of the iron, causing it to rust destructively.

Until now, a "standard" explanation for iron corrosion is that it is an electrochemical reaction, tiny local areas on the surface of the metal being assumed to act as anodes and cathodes under the influence of an invisible liquid film of water, generating minute electric currents that corrode the iron. The new theory suggests that something more fundamental takes place in the iron, even though an electrochemical reaction may also occur.

In a series of experiments that eliminated the conditions required for electrochemical reactions, the complex rusting of iron was reduced to its simplest atomic processes. Pure iron wires about as thick as a fine sewing thread were reacted with oxygen and water vapour at high temperatures [835° F. (445° C.)] under closely controlled, idealised conditions. The results of the minute-scale corruptions were studied under an electron microscope capable of magnifying objects up to 333,000 times. These studies show that with dry oxygen the iron forms a protective oxide coating from which grow billions of tiny round oxide whiskers less than one-millionth of an inch in diameter and 30-millionths of an inch high. Each whisker grows from a single, specific growth site on the wire's surface, much as individual seeds sprout from the ground into separate plant stems.

The wires were then reacted under identical conditions except that water vapour was substituted for the dry oxygen atmosphere. The moisture produces a startling change in the oxide surface. From the growth sites erupt thin, pointed, blade-shaped platelets of iron oxide that spread across the metal surface. Shaped somewhat like blades of grass, they are about one-millionth of an inch thick, 30-millionths of an inch wide and 300-millionths of an inch high. As they grow in size, they spread more than 50 times in area over the sites observed for dry oxygen alone, reaching a density of nearly one billion per square inch of surface area. The amount of iron rust they represent is 250 times that which forms when the water vapour, and the hydrogen ions it releases, are absent from the reaction. Less than one part of water vapour in 200 parts of the dry oxygen atmosphere will cause the blade-shaped crystals to form; at room temperature this would correspond to a relative humidity of about three per cent.

"We conclude that the hydrogen ions in the water vapour enlarge the areas of chemical reaction between the oxygen and iron and bring about the metal's greatly increased corrosion," Dr. Gulbransen declared. "At the lower temperatures at which iron usually rusts, this basic reaction is masked. Complex corrosion products are formed and are not stable, and the corroding metal is often removed from the reaction site.

"These experiments have enabled us to get closer to the fundamental process involved in iron corrosion and, we think, have given us new insight into its mechanism. Once such basic understanding is at hand, we should be able to do a much better job of controlling corrosion. Already we can identify two control measures which must be considered in addition to any electrochemical effects that are involved in iron rusting: hydrogen must be prevented from entering the metal; and the growth of the localised reaction sites must be inhibited by the addition of suitable alloying elements to the iron.

"The corrosion of iron has been the subject of much speculation and many scientific observations over many years. For example, we have long been intrigued by the Iron Pillar of Delhi in India, which stands in a dry climate and has resisted extensive corrosion for more than 1,600 years. We may be approaching a true understanding of its remarkable corrosion resistance. Eventually we may be able to apply that knowledge to duplicate artificially its behaviour among structures not quite so fortunately placed throughout the world."

### Jessop-Saville Move

A MAJOR move from their factory at Sheffield to new premises in Rotherham, carried out with the precision of a military operation to keep loss of production to a minimum, has been successfully completed by Jessop-Saville (Small Tools), Ltd., manufacturers of Cutanit cemented tungsten carbide tips and tools and high speed steel butt welded tools. Their new address is: Eastwood Works, Fitzwilliam Road, Rotherham (Tel. No.: Rotherham 77171-4). Additional new plant and machinery is being installed in the Rotherham factory, allowing for considerable production expansion.

### The Judgelite Company

THE JUDGELITE COMPANY has been formed to take over the plant, goodwill and stocks of what was formerly the architectural panelling division of Ernest Stevens, Ltd., of Cradley Heath, Staffs., and, in particular, the manufacture of Judgelite architectural panels in vitreous enamelled aluminium. The plant which, it is understood, is the largest of its type in the United Kingdom and includes a 20 ft. 250 kW. continuous furnace for vitreous enamelling aluminium, is being extended to cope with a full order book and to embrace an even wider field of activity. The same labour force, technical staff and associated personnel have been retained by the new company to ensure maintenance of the product's high standards in manufacture and service.

## News in Brief

**SHEEPBRIDGE ENGINEERING, LTD.**, has purchased for cash the issued share capitals of Purfection Engineering Co. (Coleshill), Ltd., and its associated company, Station Engineering Co. (Coleshill), Ltd., makers of splines, gauges and all forms of precision ground tools and gauges, and of Fulmer honing machines (under licence) respectively.

**HONEYWELL CONTROLS, LTD.**, the automation equipment company, will nearly double the size of its Lanarkshire factory over the next three years. Work on the extension will begin immediately. The expansion will give work to another 1,000 people, increasing the total labour force to 3,000.

**THE GENERAL ELECTRIC CO., LTD.**, has received an order valued at over £176,000 for six 2,150 h.p. booster pump motors and four 4,750 h.p. standby variable-speed boiler feed pump motors with liquid controllers for Blyth "B" generating station of the Central Electricity Generating Board. The equipment is being made to the order of Hathorn, Davey & Co., Ltd., who are building the pumps.

**ASSOCIATED ELECTRICAL INDUSTRIES, LTD.**, has received an order from the Central Electricity Generating Board for four 75,000-kVA. 3-phase transformers for Templeborough sub-station. The transformers will step down the 275-kV. supergrid voltage, providing a 33-kV. supply to six 40-MVA. arc-furnace transformers to be installed by the Steel, Peech & Tozer branch of The United Steel Cos., Ltd., as part of their £10 million scheme. (The two furnace transformers so far ordered are also being manufactured by A.E.I.).

THE longest single piece of graphite for its cross-section ever made is a 30 ft. column measuring 12 by 14 in. on the sides. The 3,700 lb. giant was manufactured by National Carbon Company, Division of Union Carbide Corporation, and is a prime example of the large graphite stock required for new metallurgical processes under development in various U.S. industries. Production of graphite of this unprecedented length required special manufacturing techniques, and new clamps had to be made to handle the long piece during the various processing steps.

**THE GENERAL ELECTRIC CO., LTD.**, has recently received an order valued at £139,000 for the supply and installation of a turbo-blower at the Cleveland Works of Dorman Long (Steel), Ltd., alongside two 90,000 c.f.m. blowers also supplied by G.E.C. The blower, which is to supply air to blast furnaces, will be of the centrifugal type, having an output at normal duty of 100,000 c.f.m. against a pressure of 35 p.s.i.g. and at maximum duty 110,000 c.f.m. against a pressure of 40 p.s.i.g.

**DRABBLE AND SANDERSON, LTD.**, the Sheffield saw makers, have been appointed sole agents in the U.K. for the sale of carbide tipped circular saws manufactured by the American Saw and Tool Co., of Louisville, U.S.A. The makers claim that these carbide tipped saw blades, stay sharp up to a hundred times longer than the conventional steel blades.

WHEN the Birfield Group was formed in 1938, largely by the amalgamation of Hardy Spicer, Ltd., and Laycock Engineering Ltd. it incorporated a relatively small

Hardy Spicer subsidiary, Bound Brook Bearings, Ltd., making graphited bearings under licence from the American Bound Brook Oil-Less Bearing Co. Now, an agreement between Birfield, Ltd., and the American company, provides for an outright purchase by Birfield of the latter, which will change its name to Bound Brook Bearing Corporation of America.

**WICKMAN, LTD.**, announce that the sole agency in the United Kingdom for the range of mining and construction equipment built by the Gardner-Denver Co., Illinois, U.S.A., has now been assumed by the Wickman Mining Division, Bottrill Street, Nuneaton.

A NEW company—Balzers High Vacuum, Ltd.—has recently been set up to handle in this country, commercially and technically, all the products of Balzers, A.G., Liechtenstein. The most important of these products are: equipment for high vacuum metallurgy and high vacuum coating, vacuum pumping systems, vacuum measuring and testing instruments, ultra high vacuum equipment, etc.

**WINSTON ELECTRONICS, LTD.**, have appointed Hawnt & Co., Ltd., 112-114, Pritchett Street, Birmingham, 6, as their Midland distributors. Hawnt & Co., Ltd., will sell and service the complete range of Winston products throughout the counties of Worcestershire, Warwickshire, Northamptonshire, Leicestershire, Staffordshire, Derbyshire, Nottinghamshire, Lincolnshire and Shropshire.

**FREDRK. JUKES, LTD.**, have been granted the world licence for the design, manufacture and sales of Pontifex dust and fume control and recovery equipment, which is entirely British in design and fabrication. Mr. C. A. Bainbridge and Mr. F. C. Caplin, who invented and developed the equipment for Pontifex, have been appointed to the board of Fredrk. Jukes, Ltd., and are in full control of future developments.

**THE GLACIER METAL CO., LTD.**, have recently extended their Manchester factory by some 10,000 sq. ft. and equipped it with a number of special purpose heavy machines to undertake the repair and manufacture of an increased range and volume of large whitemetal plain bearings. Bearings from 1 in. to 5 ft. bore diameter can now be manufactured.

**HIGH DUTY ALLOYS, LTD.**, announce that the telephone numbers for their Forging Division are now Redditch 4211 (day) and Redditch 4162 (night).

**ALCAN ENFIELD ALLOYS, LTD.**, and Metal Closures Group, Ltd., have reached an agreement in principle whereby Alcan Enfield will lease on a long term basis the aluminium alloy smelter and zinc alloy production facilities at London Colney, Herts., owned by John Dale, Ltd., a member of the Metal Closures Group. John Dale will continue to own and operate their light alloy foundry and continuous strip casting plant, also situated at London Colney, which will not be affected by this arrangement. Alcan (U.K.), Ltd., will market the alloys produced by Alcan Enfield Alloys, Ltd., at London Colney as well as at Bradford.

**PIONEERS** of the vacuum flash evaporator in Britain, Richardsons Westgarth have just despatched to the Sheikdom of Abu Dahbi, on the Trucial Coast, a fully self-contained sea-water evaporator which will produce 14,400 gallons of drinking water a day to replace supplies brought in by dhow at considerable expense.

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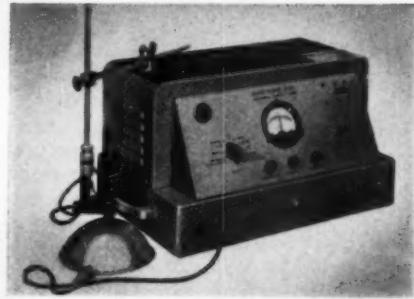
very wide range of surface finishes. Each method has its advantages and limitations, but Nash & Thompson as suppliers of the most comprehensive range of thickness measuring instruments in Britain will give you really unbiased advice to enable you to select the most suitable instrument for your particular application.

**1. BNF Plating Gauge.** For non-destructive measurement of nickel on steel, or brass, and zinc based die castings. Range 0.002 in.-0.00025 in., accuracy  $\pm 0.0001$  in. Also used as metal sorter for differentiating between similar metals. Basic price £238

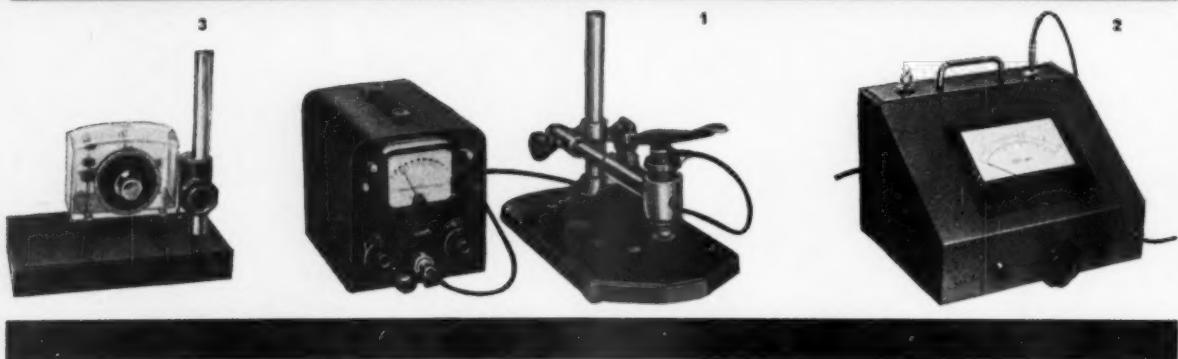
**2. Accuratest Thickness Tester.** For non-destructive measurement of non-conducting coatings on non-ferrous bases. Range 0.0001 in.-0.005 in. Accuracy  $\pm 10\%$ . Basic price £133

**3. Magne Gauge Thickness Tester.** For simple and quick non-destructive measurement of wide variety of coatings on magnetic basis metals. Accuracy  $\pm 10\%$ . Basic price £117

**4. Kocour Thickness Tester.** For destructive measurement of an exceptionally wide range of coatings and basis metals. Coulometric principle—small area of plating is stripped with standard solutions, a mechanical read-out counter stopping when all coating is removed. Basic price £246



4



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# NEWS AND ANNOUNCEMENTS

## I.S.I. American Meeting

THE Council of the Iron and Steel Institute has accepted with pleasure an invitation from the Metallurgical Society of the American Institute of Mining, Metallurgical, and Petroleum Engineers (A.I.M.E.) to visit the U.S.A. in the autumn of 1961. The American Iron and Steel Institute are kindly helping with the planning of the technical programme and with the arrangements for the visits to iron and steel works and laboratories.

The provisional programme provides for an official welcome in New York City on Thursday, 19th October, 1961, but it is anticipated that some members will arrive a day or two earlier. Members will visit important works within convenient distance from New York. They will then travel to Niagara Falls, and visit laboratories and works in the district in Canada, as well as in the U.S.A. Members have been invited by the Board of Directors of the American Society for Metals (A.S.M.) to attend the National Metal Congress in Detroit and to be guests at the annual banquet of the A.S.M. on 26th October. The itinerary will include Cleveland, Pittsburgh, Bethlehem and Washington. Opportunities will be provided for members to visit iron and steel works and laboratories in various centres. A special programme for ladies is being prepared.

At the conclusion of the tour, arrangements can be made for members to spend a few days sightseeing, visiting Philadelphia and Princeton or Colonial Williamsburg and Charlottesville. Extensions will also be arranged to enable members to visit works in the Chicago district.

## Die Casting Discussion Group

NEARLY 200 die casting engineers, mainly from the Midlands, met at the Birmingham College of Technology on Tuesday evening, 18th October, to discuss the technique of gating and venting pressure die castings. This meeting, the first of four to be held by the Zinc Alloy Die Casters Association this winter, was under the chairmanship of Mr. Reg. Turner\* and the discussion was opened by Mr. Frank Claxton†, who was assisted by Mr. J. H. Newman‡ and Mr. R. Bryan.\*

Much has been written on methods of gating die castings, and the meeting took as a basis for its discussions the paper on gating presented by Mr. Ralph Wilcox, of the American Smelting & Refining Co., Ltd., at the Third International Die Casting Conference at Stresa. The discussion, however, quickly showed that while the traditional methods of gating are well-known and widely adopted, much thought is being given to completely new ideas. Venting methods, direction of metal flow into the die and gating for vacuum die casting came in for particular attention. There is little doubt that the meeting provoked much thought on gating techniques and will lead to some of the fresh ideas discussed being tried out.

The large attendance at this meeting is further evidence of the very keen interest being shown in the development of die casting. Future meetings will be

held alternately in London and Birmingham and details can be had from the Zinc Alloy Die Casters Association, 34 Berkeley Square, London, W.I.

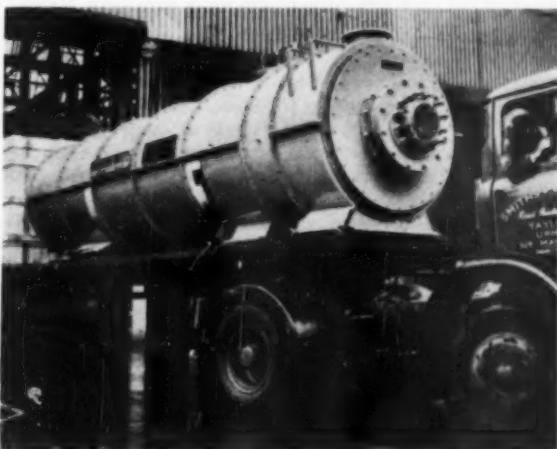
## Microanalyser Brings in Dollars

On Thursday, 3rd November, 1960, the *Queen Mary* sailed for New York carrying a piece of British-made electronic equipment which is worth over \$50,000 to Britain. This instrument, the first of three which will soon be working in the United States, was destined for the Illinois Institute of Technology in Chicago, and the other two, ordered by United States Steel Corporation and Crucible Steel Company of America, respectively, were to follow at intervals of two or three weeks. So far, four of these instruments have been ordered by organisations in the United States. These orders were secured against strong American competition, and were won largely because of a new British scanning technique.

The instrument is a good example of co-operation between pure research, the British instrument industry and the instrument user. The first scanning microanalyser was developed by the famous Cavendish Laboratory at Cambridge, in conjunction with Dr. Melford and his colleagues at Tube Investments Research Laboratories at Hinxton Hall, and the instrument is being currently produced at Cambridge Instrument Company's Cambridge works. Already, several of these microanalysers are installed in Britain.

## Foundry Foreman Training

THE continued success of the foundry foremen's training courses held during recent years has encouraged the Council of the Institute of British Foundrymen to organise a further course which will be held from Thursday, 20th April, to Saturday, 22nd April, 1961, at the Grand Hotel, Scarborough. The programme of the new



Leaving for Vienna is this hot blast heater designed and made by The Incandescent Heat Co., Ltd., Smethwick. It will supply 3,000 st. cu. ft./min. of blast air at 500° C. for a pair of 6 ton/hr. cupolas in an Austrian foundry producing radiators for central heating systems.

\* Metal Castings, Ltd.

† Wolverhampton Die Casting Co., Ltd.

‡ Birmingham Aluminium Casting (1903) Co., Ltd.

course is designed to give practical guidance to foremen and will cover such topics as: union agreements and procedure; production control and planning; health and safety in foundry work; cupola control; non-ferrous furnace control and melting practice; casting production methods; and pneumatic, hydraulic and mechanical gadgets in the foundry. Further details and forms of application to attend the course may be obtained from the Secretary, The Institute of British Foundrymen, 14 Pall Mall, London, S.W.1.

### Furnace Builders' Conference

A SUCCESSFUL conference of the Society of Furnace Builders held in Jersey from 15th to 17th October was particularly well attended by representatives of most of the leading furnace manufacturers in Great Britain. Amongst them were the firms responsible for the installation of the melting, heating and heat-treatment furnaces for the major schemes embarked on by the steel industry in recent years.

At the conference, discussions were held on the future of the industry in relation to industrial trends and developments, the use of modern techniques such as closed circuit television, the training of future personnel, the association of the Society with research organisations, and particularly with the British Iron and Steel Research Association in its investigations upon the operation of forge furnaces.

At the official dinner, the chairman, Mr. C. G. Pettit, on behalf of the members, made a presentation to the new president, Mr. H. Southern, in appreciation of his work for the Society from the time he took office (as chairman) sixteen years ago. The chairman also had the pleasure of making a presentation to Mrs. Southern. In reply, the president expressed his appreciation of the help he had received from members in the development of the Society to its present position. In his speech, Mr. Pettit referred to the changing pattern of industry, and to the exacting requirements of engineers and metallurgists in the performance of modern industrial furnaces.

### Symposium on Industrial Vacuum Plant Usage

FOLLOWING a recommendation by the Joint British Committee for Vacuum Science and Technology, the Council of the Institution of Mechanical Engineers have agreed to sponsor a symposium of papers on user experience of large-scale industrial vacuum plant, to be held at the Institution on 1st and 2nd March, 1961. The main objective of the symposium is to bring together designers and users of vacuum plant to discuss their problems and techniques. It is planned to discuss some twelve papers, which will be preceded by a general review paper. Aspects to be covered include vacuum processing and treatment of metals, steam ejection vacuum plant, capacitor drying, food processing and medicine.

### Foundry Apprentices' Competition

THE INSTITUTE OF BRITISH FOUNDRYMEN is organising a national foundry apprentices' competition open to candidates who have not attained the age of twenty on 31st December, 1960. More than half the total marks will be carried by a practical test on moulding and core-making lasting up to eight hours: there will also be written tests on foundry technology, calculations and

drawing. Candidates should have obtained a City and Guilds intermediate (now ordinary) certificate in foundry practice, or an equivalent certificate, or have received suitable technical training.

On the results of this competition will be based the selection of three apprentices to represent this country in the European Foundry Apprentices' Competition in 1961. Further particulars of the national competition and nomination forms for prospective candidates may be obtained from the Secretary, The Institute of British Foundrymen, 14 Pall Mall, London, S.W.1, to whom completed forms should be sent by 31st December, 1960.

### Induction Heating Course

DUE to the very large response to the last Induction Heating Course organised by the Process Heating Division of Pye, Ltd., in November, a number of applications for the next course, to be held on 7th and 8th February, 1961, have already been accepted. This course is free of charge and will again be held at the Globe Hotel, Cambridge. The following subjects will be covered: the fundamentals of induction heating, methods of application, the basis of coil design, hardening, annealing and tempering of small parts, brazing, soldering and processing of conducting materials. Further details may be obtained from Mrs. E. Raeburn, Pye Process Heating, 28 James Street, Cambridge.

### Physical Society Exhibition

As a result of the recent amalgamation between The Institute of Physics and The Physical Society, the Physical Society Exhibition of Scientific Instruments and Apparatus will be known in future as The Annual Exhibition of The Institute of Physics and The Physical Society. In 1961 it will be held at the Royal Horticultural Society's Old and New Halls, Westminster, London, S.W.1, and will be open to non-members from 2 p.m. on Monday, 16th January, to 1 p.m. on Friday, 20th January. The exhibition will open at 10 a.m. each day from Tuesday to Friday and will close at 7 p.m. from Monday to Thursday (Tuesday 9 p.m.). Tickets are available from The Institute of Physics and The Physical Society, 47 Belgrave Square, London, S.W.1.

### Refractories Film

"The Steetley Story" is a film about refractories and about the largest producer of refractory materials in Western Europe and the Commonwealth—The Steetley Co., Ltd., of Worksop, Nottinghamshire. Wherever fire and furnaces are used in the service of mankind to produce steel, glass, cement, gas and coke, to name but a few, refractories are called upon to contain and control the enormous quantities of heat involved. The refractories field of application is widespread, ranging as it does from the simple firebrick found in every household fireplace, to the vital materials used in earth satellites and atomic power plants: one of the most important applications is in the lining of steel furnaces.

The film opens with a view from the air of one of the Steetley plants. The helicopter from which the scene is being filmed, slowly descends until we approach the building containing the administrative offices. Here we meet Christopher Chataway, who introduces the film and speaks the commentary. From here, the film

illustrates the acquiring of raw materials and the many processes associated with the manufacture of refractory products.

One of the most interesting features of the film is that illustrating the sea-water magnesia plant at Hartlepool. Here is produced refractory magnesia from dolomite (magnesian limestone) and sea-water, a process which was pioneered by The Steetley Co. in the late 1930's (from an experiment made in the laboratory using an ordinary bath-tub and six foot tube as a miniature rotary kiln). The Hartlepool plant is the only one of its kind in the United Kingdom, and is also the largest in the world, despite the substantial works subsequently built in the United States.

From a tour of some of Steetley's many plants in this country, the scene moves to Canada, where the company operates a healthy Canadian division. In addition to refractory materials, the Canadian subsidiaries quarry dolomite stone for many purposes including buildings, some of which are seen in the film. Back in Britain, we see some of the uses of Steetley's products and the film ends with a brief summing up by Christopher Chataway.

The film is available on loan in 16 mm. or 35 mm. Eastmancolour, on application to The Steetley Co., Ltd., Gateford, Worksop, Notts.

### Course on Failures

A COURSE of six lectures will be held at the Institute of Technology, Bradford, at 7 p.m. on successive Wednesday evenings, commencing on 18th January, 1961. The course will commence with a general lecture on types of failure, and fatigue will be dealt with subsequently in detail. Other lectures will emphasise the types of failure which predominate in various branches of engineering. Application forms, giving full details, may be obtained from the Registrar, Institute of Technology, Bradford, Yorks.

### Foseco Companies Change Name

In view of the Foseco group's expansion of interests and activities beyond the foundry industry, particularly to other metallurgical processes, it has been felt desirable to have a change of name for each of the two principal controlling companies of the group. The holding company, hitherto known as Foundry Services Holdings, Ltd., and in which the controlling interest is held by a public company, Minerals Separation, Ltd., becomes Foseco Holdings, Ltd., and the managing company, hitherto known as Foundry Services International, Ltd., becomes Foseco International, Ltd.

### Steel Extrusion Plant Agreement

An agreement which is expected to foster the use of steel extrusion plant has been concluded between Mannesmann-Meer A.G., of Mönchen Gladbach, Germany, and The Loewy Engineering Co., Ltd., of Bournemouth. The subject of the agreement, which covers the whole world, is the supply and installation of plant for the production of seamless tubes of carbon steel and low alloy steels. Mannesmann-Meer are well known as suppliers of stretch reducing mills and related auxiliary equipment for tube manufacture and Loewy Engineering has been building steel extrusion plant for many years.

The agreement provides for technical co-operation, an

economic exploitation of the manufacturing facilities which are at the disposal of the two companies, and includes the services of Mannesmann-Meer Inc., of Youngstown, Ohio, who for several years have been dealing with Mannesmann-Meer equipment in the United States. The experience of both companies in the production of seamless tubes is at the disposal of prospective customers and big schemes for integrated plant in many countries are under discussion.

### Electrical Copper Symposium

ON 19th October, 1960, at the Dorchester, London, some 200 members of the electrical industry attended a symposium arranged by the Copper Development Association. Under the chairmanship of the Earl of Verulam, four papers were presented:—

"The Economic Use of Copper in Power Transformers," by F. W. Gee (Ferranti, Ltd.).

"Cooled Copper Conductors for Electrical Equipment," by G. W. G. Cogle, E. R. Hartill and J. Tudge (Associated Electrical Industries (Manchester), Ltd.).

"Some Notes of the Use of Copper as a Material for the Construction of Waveguides," by L. E. Hall and B. H. Meggs (Associated Electrical Industries, Ltd., Electronics Apparatus Division).

"Copper Alloys for Conductivity Applications," by T. L. Richards (Copper Development Association).

In the discussions which ensued, both manufacturers and users of the equipment and materials concerned were usefully able to take advantage of this opportunity to present their individual problems or requirements as applied to various specific applications.

A display of various representative items demonstrated the important role which copper plays in the electrical industry and particularly in those fields covered by this symposium.

### Metallurgical Research at High Pressures

DR. J. E. HILLIARD, of the Research Laboratory, General Electric Company, Schenectady, U.S.A., is to give a lecture on "Metallurgical Research at High Pressures," under the auspices of the Metal Physics Committee of the Institute of Metals. The meeting will take place at the Institute's Headquarters, 17, Belgrave Square, London, S.W.1, on Thursday, 12th January, 1961, at 6.00 p.m. Visitors are welcome: tickets are not required.

### Inco Iron Ore Expansion

THE INTERNATIONAL NICKEL CO. OF CANADA, LTD. is to expand its iron ore recovery plant at Copper Cliff, Ontario, tripling its capacity. Using a process invented by the company's research staff and successfully established in its existing iron ore recovery unit, the expanded plant will treat 1,260,000 short tons per year of nickeliferous pyrrhotite high in iron content. The process involves solid state pyrometallurgical operations, and the removal of nickel by atmospheric pressure leaching, in high-capacity units of novel design.

The diversion of this large quantity of pyrrhotite to the expanded iron ore recovery plant will effect a 40% decrease in the tonnage of material which otherwise would have to be handled by the nickel section of the Copper Cliff smelter. The change in practice in the nickel



**Besides heating forging billets by induction, Garringtons Ltd., Bromsgrove—one of the largest manufacturers of forgings in Europe—manufacture electrical induction heating equipment in a separate division. Delegates to the recent British Electrical Development Association Industrial Conference are here seen viewing special purpose equipment of this type during the course of a visit to Garringtons.**

smelter will result in important smelting economies and provide a basis for further major advance in nickel extractive metallurgy. In the iron ore recovery plant, capacity for the output of iron ore pellets containing 68% iron, which have established a high reputation for quality, will be raised to 900,000 short tons per year, and more nickel will be recovered from the same amount of pyrrhotite than is now possible in the nickel smelter.

The expanded plant is scheduled to be in full operation in 1963. It will entail a total estimated capital outlay of \$50,000,000, of which up to \$10,000,000 is scheduled for 1961. The project is another milestone in the company's intensive research efforts to achieve maximum utilisation of the many elements found in its Canadian ores. The company is confident that the strong and growing demand for high-grade iron ore, particularly in the United States, will assure an outlet for the entire tonnage of Inco's premium iron ore pellets.

## Personal News

**MR. A. S. GILL** has retired from the board of George Cohen Sons & Co., Ltd., on attaining the age of 65, and will practise as an industrial consultant.

**MR. P. JARDINE**, group financial controller, Metal Industries, Ltd., has been appointed to the boards of two Metal Industries subsidiaries: J. G. Statter & Co., Ltd., switchgear manufacturers, and Minerva Mouldings, Ltd., manufacturers of plastic mouldings, both of Amersham, Bucks.

**MR. E. V. BEATSON**, of Joseph Lucas, Ltd., has been appointed to the Council of the British Welding Research Association to serve on the Association's research board. His principal fields of interest are resistance welding and brazing.

THE United Steel Cos., Ltd., announce that Lt.-Cdr. G. W. WELLS, D.L., managing director (production) of the company, and director and general manager of the Appleby-Frodingham Steel Co. branch, becomes chair-

man of Appleby-Frodingham Steel Co. Mr. J. D. Joy, director and deputy general manager of Appleby-Frodingham, becomes director and general manager of the branch.

THE L. S. Starrett Co., Ltd., the Jedburgh makers of precision tools and measuring instruments, have appointed Mr. J. BELL chief engineer and Mr. S. CROSSEY hack and bandsaw superintendent. Mr. Bell was formerly with Giddings & Lewis-Fraser, Ltd., and Mr. Crossley was with Arthur Balfour & Co., Ltd. On the sales side the following representatives have been appointed: Mr. G. H. BARCLAY (Scotland), Mr. A. Swancott (North Midlands) and Mr. H. GAUNT (South Midlands).

DR. T. B. TOMLINSON, who joined Southern Instruments, Ltd., as chief engineer in March of this year, has also taken over responsibility for design and development in the newly-formed Drayton-Southern, Ltd.

THE constitution of the board of directors of the newly-formed Brooke Tool Automation, Ltd., is as follows: Mr. A. G. B. OWEN, C.B.E., J.P. (chairman), Mr. H. S. HOLDEN (managing director), Mr. H. H. EVANS (production manager), Mr. E. W. HANCOCK, Mr. E. W. B. OWEN, and Mr. K. G. WALTON (general manager).

MR. J. IRELAND has been appointed general manager of the Cheadle Heath plant of British Driver-Harris Co., Ltd., with Mr. J. COOPER as works manager. Two new assistant works managers have also been appointed: Mr. A. HESSION (wire) and Mr. G. CASSON (strip).

MR. E. M. THOMAS has been appointed resident representative in Hull of the machinery division of George Cohen, Sons & Co., Ltd. He succeeds Mr. H. WHYMAN, who has for some years been well known in industry throughout the area and who has now been appointed manager of the division's Kingsbury branch. Mr. Thomas has taken up residence at 79 Eppleworth Road, Cottingham, Yorks. (Tel.: Cottingham 47263.)

MR. A. E. BELL and MR. R. LANCASTER have been appointed to the board of Distington Engineering Co., Ltd., a subsidiary of The United Steel Cos., Ltd. Mr. Bell becomes director and commercial manager, and Mr. Lancaster becomes director and general works manager.

FIVE new directors have been appointed to the Board of John B. Pillin, Ltd., of West Hartlepool, makers of Lubquipment and a member company of the Castrol group. They are: Mr. R. ADAMS, Mr. J. A. P. COE (secretary), Mr. L. J. FIELD, Mr. A. F. MACDONALD (works manager) and Mr. J. W. MACMAHON. Mr. MacMahon is also a director of the group's industrial company, Wakefield-Dick Industrial Oils, Ltd., and general manager of that company's industrial division. Mr. Adams and Mr. Field are executives at the group's headquarters in London. The appointment of two additional directors to the board of Castrol (Ireland), Ltd., has also been announced. They are Mr. L. A. COURTNEY and Mr. E. E. HUGHES, both of whom hold executive positions at Castrol's London headquarters. Mr. Courtney is manager of the agricultural department and Mr. Hughes is assistant manager, Castrol division.

FOLLOWING the retirement of Mr. J. ABBERLEY, the board of Firth Cleveland Tools, Ltd., announce that Mr. J. C. ABBERLEY has been appointed director in charge of their Tipton works, and Mr. A. H. McILREATH director in charge of their factories at Wolverhampton.

# RECENT DEVELOPMENTS

## MATERIALS : PROCESSES : EQUIPMENT

### Metal Finishes

THE new Parker 45 finishes are quick air-drying materials applied at room temperature by dip or spray, and are intended for use on Bonderized or Parkerized surfaces. Parker Finish P.45C is designed to give a clear film, while Parker Finish P.45B gives a uniform black surface. They have fairly low viscosity and may subsequently be over-painted if required.

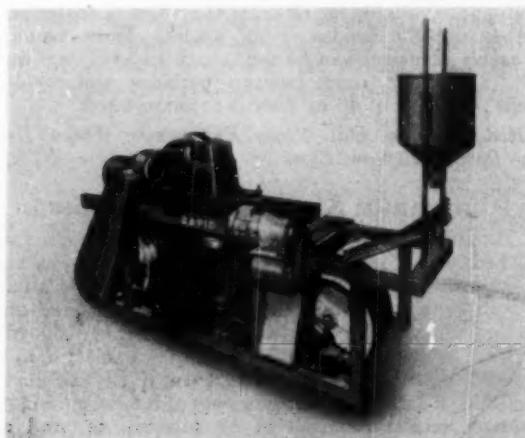
Both finishes are formulated to give a slight reaction with the underlying phosphate coating or bare steel surface so as to give maximum possible adherence and sealing value whilst, at the same time, leaving a minimum film thickness which is both tough and flexible. They give a high degree of corrosion protection on bare surfaces although, of course, the protection is much superior when they are applied to a Bonderized or Parkerized surface.

Generally speaking, Parker P.45 finishes are used as supplied, although thinners are available for both products. When the finishes are applied by dipping, they should be contained in a mild steel tank with a tightly fitting lid, and after dipping the work at room temperature, the finish air dries to 'touch dry' in approximately 10 minutes and dries completely in approximately 30 minutes. The rate of drying P.45B and C can be speeded by stoving the parts for approximately 10 minutes at 200-250°F. When the Parker Finish P.45B and C are applied by spray, normal techniques are employed.

*The Pyrene Co., Ltd., Metal Finishing Division,  
Great West Road, Brentford, Middlesex.*

### High Intensity Magnetic Mineral Separator

THE latest addition to the range of magnetic mineral separators made by Rapid Magnetic, Ltd., is a single disc unit which treats granular free-flowing minerals such as garnet, monazite, ilmenite, wolfram, columbo-tantalite etc., and incorporates a magnetic scalper prior to the



disc which removes highly magnetic minerals such as magnetite, ferro-silicon and other iron oxides, so preventing agglomeration at the disc discharge. By mechanical adjustment of the disc and rheostatic control of the magnet current, a further two magnetic minerals are separately recovered in one pass and discharged either side of the separator belt. Clean non-magnetic materials are discharged at the conveyor head. Feed is by gravity from a hopper; alternatively a vibratory unit is available. The capacity is dependent on the characteristics of the material and degree of separation required.

Industrial applications include the removal of fine iron contamination from abrasives, refractories, chemicals, dehydrated foods, etc., where a completely iron-free product is essential. The makers will freely execute preliminary tests on representative samples.

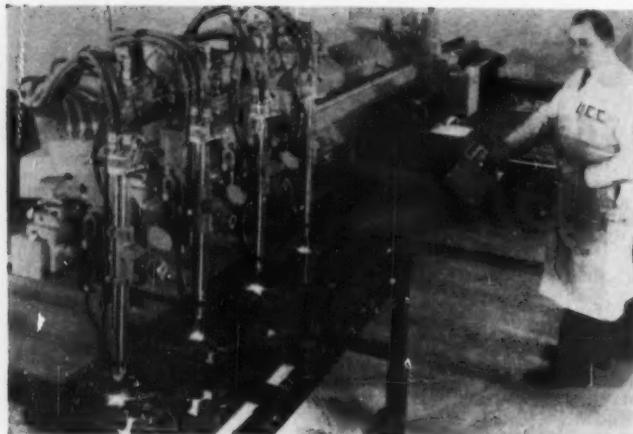
*Rapid Magnetic, Ltd., Lombard Street, Birmingham 12.*

### Shape-Cutting Machine

THREE new dual-carriage shape-cutting machines featuring a unique drive principle are now available from Union Carbide International Company, Division of Union Carbide Corporation. Known as "roller drive," the patented drive principle of the new Oxweld CM-56 machine completely removes the drive wheel from the template table. In ordinary cutting machines the drive wheel is on the tracing table surface riding on the template. With the new CM-56 it is not necessary to provide protection for the template to prevent scuffing or damage to the outline to be traced. There is no force tending to shift the position of the line drawing and it is easier to move and align the template. Drawings can be positioned without raising the tracing head.

A main rail carriage running on the main rails carries another pair of rails supporting the transverse carriage. With the combined motion of the two carriages it is possible to follow and cut metal parts and forms of any shape. Either carriage on the CM-56 can be locked, making it possible to produce perfectly straight and accurate cuts parallel to the main rails or at exactly 90° to the main rails.

The Oxweld CM-56 is equipped with Union Carbide's new photocell tracer with built-in kerf adjustment. Simple pencil or ink sketches of intricate metal shapes and forms are used to guide the CM-56. The new Linde photocell tracer accurately follows sketches on ordinary paper, eliminating the need for expensive metal or plastic templates, photographic negatives or complicated and costly silhouettes. Tracing accuracy is  $\pm 0.005$  in., and ordinary light, sunlight or shadows do not interfere with the precision operation of the tracer. The tracer can negotiate 90° turns with  $\frac{1}{8}$  in. radius at speeds up to 16 in./min. and 90° turns with  $\frac{1}{4}$  in. radius at speeds up to 27 in./min. The CM-56 may be located indoors or outdoors without regard for lighting conditions. The operator can use as much light as he needs to follow the cutting operation without fear of upsetting the cutting pattern, he can steer



manually or use remote control buttons to engage the template.

Parts can be reproduced from an exact size drawing without the necessity of making allowance on the drawing for kerf width (metal removed by the cutting oxygen jet). Drawings of parts are made or traced in the exact size of the part desired, and the kerf compensator dial is then set for the plate thickness being cut. In this way it compensates for kerfs up to  $\frac{1}{4}$  in. wide so that reproduced parts are exactly the same size as the line drawing.

The 10 ft. model carries up to eight torches, the  $\frac{7}{2}$  ft. model up to six, and the 5 ft. up to four. Preheat gases and cutting oxygen are controlled by solenoid valves large enough to assure an adequate gas supply for multi-torch operations. Each torch has an individual cutting oxygen solenoid valve to assure instantaneous shut-off of gas, eliminating overrun at the end of a cut. Valves are controlled by electric switches on the control panel. In addition each torch has individual valves for fine adjustment of the preheat gases and a shut-off valve for the cutting oxygen. Motorised torch holders provide automatic up and down adjustment of all torches, and a new automatic height adjustment control is also available to deal with the slightest deviation in level of the plate surface.

*Union Carbide International Company, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, New York, U.S.A.*

### Portable Blast Cleaner

A COMPLETELY self-contained portable blast cleaning unit that is no bigger than a small hand vacuum cleaner is now being produced by Hodge Clemco, Ltd. Known as the Educt-o-Matic, it weighs only seven pounds and contains its own vacuum pick-up and dust extraction unit, so that the only connection required is an airline. This should be  $\frac{1}{2}$  in. internal diameter, supplying 33 to 60 cu. ft./min. at a pressure of 90-100 lb./sq. in. There are no electrical connections.

The Educt-o-Matic is invaluable in areas where open blasting cannot be done. It can be used, for instance, in welding shops for cleaning welds or for cleaning before welding; in areas where dust would be a nuisance, such as in canneries, motor car factories and chemical plants;



in highly mechanised factories; and in areas that are difficult of access. It is small enough to fit through tank manholes, and the blast head can be swivelled for use at any angle.

The abrasive is continuously recirculated so that there is very little wastage. A full charge of four pounds will normally blast for 15-40 minutes, according to the skill of the operator, although very little skill is required for maximum efficiency. Compressed air sucks the abrasive from its container into a venturi-type nozzle which blasts the air/abrasive mixture perfectly evenly over the area enclosed by a rubber boot. This gives a blast pattern of  $1\frac{1}{2}$  in. diameter. A vacuum picks up the abrasive with the material blasted from the surface and passes them to a small cyclone where they are separated, the removed material being blown into a dust bag for later disposal. The standard rubber boot attachment can be used for cleaning flat or large diameter surfaces, welds, floors and ceilings. Alternative attachments are available for cleaning the edges of plates and shapes and the insides of  $90^\circ$  angles. Most common re-usable abrasives can be used, such as steel shot and grit, aluminium oxide, silicon carbides and walnut shells: a mesh of 40 or finer is recommended.

*Hodge Clemco, Ltd., Victor Engineering Works, New Road, Rainham, Essex.*

### Aluminium Alloys for Shipbuilding

ALCAN INDUSTRIES, LTD., formerly Northern Aluminium Co. Ltd., have recently put on the market an improved aluminium-magnesium alloy, Noral D54S, which is now offered as a standard material for both plates and sections for structural use to the requirements of Lloyd's Register of Shipping. The minimum mechanical properties laid down by Lloyd's Register for aluminium structural alloys are a 0.1% proof stress of 8 tons/sq. in., an ultimate tensile strength of 17 tons/sq. in. and an

elongation of 12% on a 2 in. gauge length. Whilst these are readily attained by plate supplied to the B.S.1477: NP5/6 specification, it has been necessary in the past to supply extruded sections to the B.S.1476:NE6 specification in order to guarantee these properties. Noral D54S, with a nominal composition of 4½% magnesium, 3% manganese and 1% chromium will meet Lloyd's requirements both in the plate and extruded form. This applies in the annealed (O) condition as well as the "as manufactured" (M) condition, leading to the further advantage that the properties after welding do not fall below the minimum properties specified for the material as supplied. Noral D54S, which is generally similar to the American alloy AA5083, falls within the range of chemical composition covered by the NP5/6 plate specification but is not yet covered by a British Standard for extruded sections.

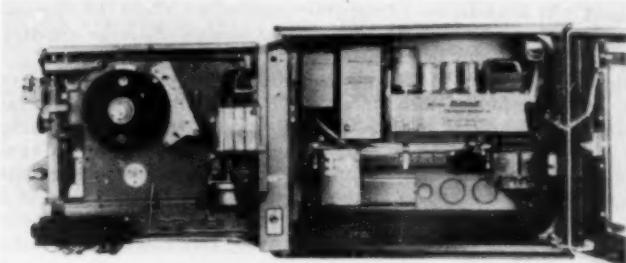
*Alcan Industries, Ltd., Banbury, Oxfordshire.*

### Modular Construction for ElectroniK Instruments

SIMPLER servicing and lower maintenance costs are achieved in a new range of Honeywell ElectroniK instruments constructed on the modular principle. Now, all major electrical components are contained in four separate quick-change sub-assemblies (modules) which provide these advantages, and also give finer performance. All these sub-assemblies, except the measuring unit, may be incorporated in existing ElectroniK instruments. The four main modules are: constant voltage unit, measuring circuit unit, sectionalised balancing motor and quick release amplifier.

The new constant voltage unit, which obviates batteries and periodic standardising, keeps the voltage across the slidewire to  $\pm 0.03\%$  for  $\pm 10\%$  line variations, or  $\pm 0.15\%$  where ambient temperature varies  $\pm 55\%$ . This is achieved by regulating the output voltage of a small A.C.-D.C. supply with cascaded Zener diodes; a high output stability is achieved without change in the instrument's accuracy. Spurious pen movement is thus eliminated, a benefit formerly only partly obtained by using a separate standardising motor. The constant voltage unit requires no maintenance.

Truly "packaged" in design, the measuring circuit unit is fully self-contained and has clip-on resistor panels for quick range changing; no soldering is involved, the change requires no specialist skill and so can be made by means of a screwdriver. Similarly, changing the actuation requires only the alteration of a spool



panel and the fixing of a new chart or scale. Improved defence against stray voltages is achieved by shielding and the use of short cable runs.

The sectionalised balancing motor comprises four easily accessible sections and any major part is replaceable within two minutes. This motor has been found specially reliable owing to the incorporation of built-in shock absorbers; it also has leak-proof oil-wick lubrication.

Any of fifteen amplifier units with quick connect plug may be fitted, providing gain of up to 40 million times for input impedances between 400 and 50,000 ohms.

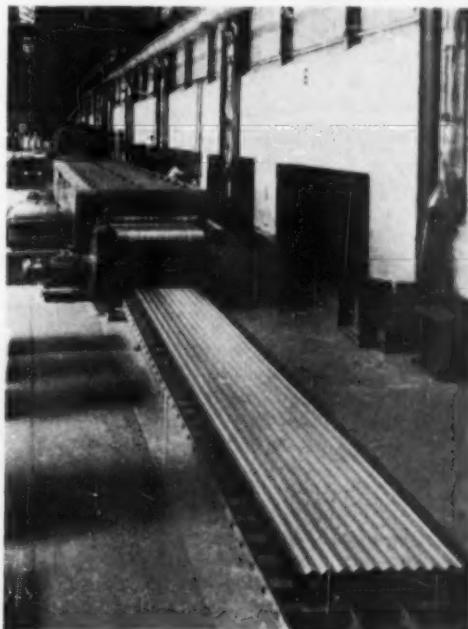
In the illustration of one of the new instruments, the constant voltage unit, measuring circuit unit, and plug-in amplifier can be seen (left to right) in the case of the instrument. The sectionalised motor is just to the left inside the swing door.

*Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.*

### Longer Galvatite Sheets

JOHN SUMMERS & SONS, LTD. have introduced into their plant new machinery, enabling them to supply extra-long Galvatite—corrugated galvanised steel sheet. This material is widely used for cladding agricultural and industrial buildings, and the introduction of these long sheets will affect economies in building. Sheets which were formerly supplied in lengths of 10 ft. and 12 ft. can now be supplied up to 22 ft. in length, in the standard 3 in. corrugation. The illustration shows a 22 ft. length leaving the corrugating machine.

Loss of material at the end laps of sheeting both on roofing and siding will be much reduced, as the long sheets span several purlin spacings. In some instances one sheet may span from eaves to ridge or from ground level to eaves. The new method of corrugating ensures

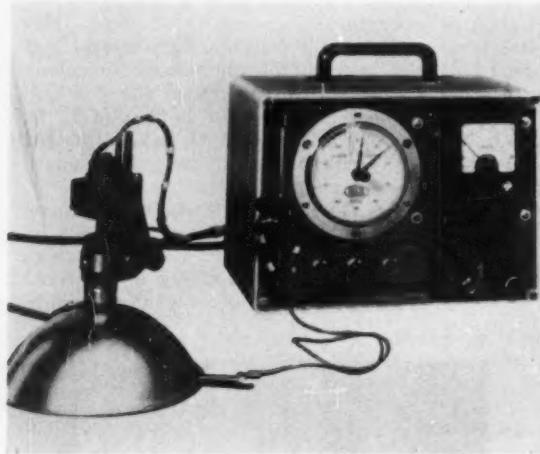


that the corrugations are perfectly parallel, making erection easier and reducing building time. The length of 22 ft. is an arbitrary figure and is imposed as a maximum length that can be conveniently handled. Longer sheets can be supplied if customers provide suitable transport.

*John Summers & Sons, Ltd., Hawarden Steelworks, Shotton, Chester.*

### B.N.F. Coulometric Thickness Meter

BASED on a design by the British Non-Ferrous Metals Research Association, this new plating meter is being manufactured by Nash & Thompson, Ltd., as an accurate means of measuring the thickness of most industrial electroplated coatings. The coulometric principle involves stripping the plating from a standard area and measuring the integration of time and current density. The operation is very simple, normally occupying only a few minutes and the meter switches itself off when the stripping action is completed. The thickness of the plating thus removed is determined from a dial reading and a multiplying factor.



This B.N.F. coulometric thickness meter is mains operated and is suitable for use in both the research laboratory and the inspection department and can be used by non-technical staff. The circuits used are transistorised and all components are carefully rated to ensure a long and reliable use under industrial conditions.

When platings are measured for thickness by this method a small, accurately determined, area of the surface is attacked by a stripping solution, with or without agitation. At the same time a current is passed through the cell and the plating and an integrating circuit provides a reading based on the integration of the current density and the time taken to strip the plating. Readings between 0.000004 in. and 0.0015 in. can be made to an accuracy of  $\pm 5\%$  on the following combinations: chromium on nickel; nickel on steel; nickel on brass; nickel on copper; copper on steel; copper on zinc; cadmium on steel; nickel on aluminium; and zinc on steel.

To take a reading, the cell on the swing arm is placed on the plated surface and a quantity of the stripping solution placed in the cell. The cell potential is set to

the correct voltage as indicated on the meter and the start control switched on. No further action is required until the stripping is completed, when the instrument automatically switches itself off. A note is then taken of the readings on the integrator dials and these are multiplied by the factor for the type of plating.

*Nash and Thompson, Ltd., Hook Rise, Tolworth, Surrey.*

### Lightweight X-ray Unit

A NEW portable X-ray unit of 150 kV. capacity and a weight of 51½ lb., has been introduced by Pantak, Ltd. The new unit, known as the Baltospot G150, is available in two models, the type D head having a 60° field, whilst the type P has a 360° field. In both units the beam angle is 35° and the head is offset. The focal spot size of the tungsten target is 1.5 × 1.5 mm. in the E150D, whilst that for the G150P is 0.8 × 2.1 mm. A particular feature of the offset head is its use, especially in the panoramic version, in the examination of closed vessels of small diameter, the examination of a 3 ft. diameter tubular vessel with a welded dished end being well within the capacity of the unit.

Sulphur hexafluoride is used for both insulation and cooling of the X-ray tube, forced circulation cooling of the anode being effected by means of an internal blower. The tankhead is of drawn aluminium with bolted dished ends and amongst the numerous protection devices fitted are shock absorbent mountings for the tube in the tank head and further shock absorbent mountings for the tank head in the cradle. A thermoswitch is fitted to guard against excessive temperature rise. The length of the tankhead is 1 ft. 9 in. and the diameter is 8½ in., the weight being 46 lb. The weight complete with end rings is 51½ lb., and the weight of the protective cradle is 6½ lb.

The control unit operates from a 220 V. 50/60 c./s. A.C. supply, using 6 A., and accommodates mains fluctuations of 10% either way. Stepless type kilovoltage and tube current controls are incorporated, giving continuously variable control. Experimental exposure charts for 16 in. and 28 in. focus/object distances are supplied in the lid. The dimensions of the control case are 1 ft. × 9½ in. × 6½ in., the weight being 27 lb.

*Pantak, Ltd., Vale Road, Windsor, Berks.*

### Oil Type Rust Preventive

CRODAFLUID 90/40 is a thin brown liquid intended for inside shop use for the protection of surfaces prior to final fabrication, and for parts being passed from one assembly shop to the next. It is not of the class of rust preventives intended to give protection against outside exposure. Indeed, its protective value under such conditions could be short—of the order of 4–7 days. It is useful as a temporary protective where the circumstances do not warrant expenditure on the more effective temporary protectives.

90/40 will wet clean metallic surfaces easily to give a uniform film of protective material. The general methods of application are dipping, spraying, brushing or swabbing and injection. Dipping has many advantages and is recommended wherever practicable. Like spraying, it can be incorporated as an integral part of a conveyor production line. Brushing or swabbing is the simplest method of application, but the coating lacks uniformity.

*Croda, Ltd., Cowick Hall, Snaith, Goole, Yorkshire.*

# CURRENT LITERATURE

## Trade Publications

WE have received from the Baker Platinum Division of Engelhard Industries, Ltd., their latest thermocouple publication, which presents the case for the use of precious metal thermocouples, stressing the advantages as regards accuracy, stability, and resistance to chemical attack or oxidation, and claiming that taking into account length of life and the high scrap value—the cost of using them does not compare unfavourably with the use of base metals. Details are then given of the range of thermocouples available from the company, ranging from the most widely used, platinum *v.* 90% platinum-10% rhodium and platinum *v.* 87% platinum 13% rhodium couples to the high temperature iridium *v.* tungsten and tungsten-rhenium *v.* tungsten couples, which can be used intermittently, in inert atmospheres or *in vacuo*, at temperatures up to 2,300° C. and 2,800° C. respectively.

THE first of two publications (R.D. 77 (Sec. 2)) recently issued by Morgan Refractories, Ltd., deals with the monolithic refractories known as mouldables, castables and ramming materials, which have long been used for repair work and have now been developed in quality to the stage where they are used as structural refractories. Particulars are given of various grades, ranging from 35% alumina up to 98% alumina.

The second leaflet (R.D. 95) describes briefly the Purox range of high quality oxide refractories—alumina, thoria, magnesia and zirconia. The use of graphite lubricants in the glass industry is featured in a leaflet (G.P. 60) issued by another Morgan company—Graphite Products, Ltd.—whose Foliac preparations are used as mould lubricants in automatic glass forming machines.

A NEW eight page publication has been produced by Henry Wiggin & Co., Ltd., on Incoloy DS nickel-chromium-iron alloy. This alloy, formerly known as Nimonic DS, though less resistant to oxidation than the higher nickel-containing alloys, is a useful material in oxidising conditions at temperatures up to 950° C., and in reducing atmospheres may sometimes be used at even higher temperatures. The publication gives details of physical and high-temperature properties and includes reference to fabrication techniques.

THE first edition of Publication No. 52 has just been released by Keith Blackman. It covers by description, drawings, photographs and capacity tables, the new range of Tornado-Cyclogal high efficiency dust collectors which are available in seven sizes, with normal rated duties from 160 to 42,500 cu. ft. of dust laden air per minute. Copies may be obtained from the Publicity Department, Keith Blackman, Ltd., Mill Mead Road, London, N.17.

Two new publications describe the standard range of vacuum furnaces made by The General Electric Co., Ltd., in association with Vacuum Industrial Applications, Ltd., and British Geco Engineering Co. Vacuum induction furnaces, dealt with in publication E.46, are suitable for melting and casting metals gas free, from crucibles ranging in capacity from one pound

to one ton. The work chamber and oil diffusion pump are mounted on one framework, the rotary pump being mounted separately to avoid vibrating the furnace. Publication E.47 indicates that "cold retort" types of vacuum resistor furnaces are available, and are suitable for the laboratory or for production. Sintering, brazing, melting and heat treatment under vacuum are typical applications for this type of furnace. In addition G.E.C. can also supply special equipments such as vacuum quenching furnaces to meet particular requirements.

BESIDES catering for the production shop, Wild-Barfield Electric Furnaces, Ltd., have established a reputation for laboratory muffles and small furnaces which are today in world-wide use. A new booklet dealing with this side of Wild-Barfield's work gives particulars of units ranging from horizontal rectangular muffles to high temperature tube furnaces, and from small gold melting furnaces to pyrometer testing furnaces and Tensometer tensile testing furnaces. Other recent Wild-Barfield publications include one dealing with gas carburising and carbonitriding processes and equipment therefor, one on shaker hearth furnaces for continuous controlled hardening, and one on the 2½ kW. induction heating equipment designed to bridge the gap between very small units and the larger industrial equipment.

A BRIEF but informative synopsis of what has been achieved by the A.E.I. Electronic Apparatus Division in advising and assisting manufacturers desirous of taking advantage of the benefits offered by modern electronic techniques is given in an illustrated publication (4051-81, Ed. A) just issued. The publication imparts an insight into what can still further be accomplished to reduce costs and increase efficiency in a great many existing and new industrial processes, and includes sections on electronic motor control, power magnestats, static switching, welding controls, voltage regulators, electronic timers, high frequency heating, photo-electric equipment, control of paper machine drives, crane approach warning system, "Clearcall" carrier-frequency communication system, safety devices, etc. Copies can be obtained on application to the Advertising Department, A.E.I. (Rugby) Ltd., Mill Road, Rugby.

A BROCHURE in colour which describes in detail six typical uses of the EMIDEC 1100 all-transistor data processing system, has recently been issued by E.M.I. Electronics, Ltd. The applications described range from production control to hire purchase accounting, and include special chapters on payrolls, and current accounting for banks. Each section is illustrated by a typical flow chart, and an ingenious throw-out flap on the back cover facilitates instant reference to the number of units of equipment required for each application described. Copies of the brochure can be obtained on application to: Computer Division, E.M.I. Electronics, Ltd., Hayes, Middlesex.

AN up to date leaflet has been issued, covering Philips stud-welding equipment, and is obtainable from the company's sole distributors in the U.K., Research and Control Instruments, Ltd., Instrument House, 207 Kings Cross Road, London, W.C.1. The leaflet (RCL

5909) gives the latest information about apparatus and accessories available, the main features of the technique and illustrates a typical application.

SINCE its foundation a hundred years ago the David Brown organisation has fulfilled a unique—and often unsuspected—role in the technological advancement of human endeavour in many parts of the world. A new publication—*David Brown: Some Facts and Figures*—is intended to indicate some of the organisation's more spectacular contributions, and to illustrate its worldwide interests and activities—the result of a century of enterprise and progress.

THE engineering group of The General Electric Co., Ltd., has recently extended its range of circular electric lifting magnets to include two new sizes of 30 in. and 80 in. diameter. Full information of these two new sizes is included in the revised edition of Technical Description No. 379. In addition to standard circular lifting magnets, G.E.C. makes many other types including those suitable for salvaging steel and iron cargo from the holds of sunken ships; for handling hot ingots up to 650° C.; for lifting coils of steel strip; and for charging rotary furnaces with operating temperatures up to 1,000° C.

A RECENT Henry Wiggin publication explains briefly the composition and available types of casting in the Nimocast series of nickel-chromium alloys. Many of the applications, which are illustrated, are components of British gas-turbine engines, such as turbine-stator blades, rotor blades, discs and rings. The publication, which gives an indication of the range of forms which can be cast in the Nimocast series of alloys, is obtainable free on request from Publicity Department, Henry Wiggin and Co., Ltd.

No. 41 in the series of monographs on organic chemical reagents issued by Hopkin & Williams, Ltd., Chadwell Heath, Essex, deals with 4:7-diphenyl-1:10-phenanthroline (Bathophenanthroline). This is a reagent for iron and information is given concerning its use for the detection and determination of iron, particularly in small amounts.

THE *Buyers' Guide* first published by the British Gear Manufacturers Association in 1955 has now been revised and reprinted. The *Guide* is designed to assist buyers of gears both at home and overseas by making known the range of products and services which are available from British manufacturers.

ABSTRACTS of papers presented at the Fifth Symposium on Magnetism and Magnetic Materials, Detroit, 1959, form an interesting feature of the September/October issue of *The Nickel Bulletin*. The remainder of the abstracts cover practically every field in which nickel or nickel-containing materials are of industrial importance. Work on methods of improving the corrosion-resistance of nickel/chromium electrodeposits (particularly in connection with the plating of car components) continues to be reflected in the Electrodeposition Section. Properties, applications, determination and analysis are the main subjects of the Nickel Section, while abstracts of papers on the properties of cupro-nickel alloys form the basis of the section on non-ferrous alloys. Items outlining the scope and salient findings of five investigations of the characteristics of nickel-containing spheroidal-

graphite irons make up the Cast Iron Section, and the abstracts in the section covering low-alloy steels reflect, *inter alia*, the growing interest in the behaviour of such materials at high and low temperatures. Over fifty of the abstracts comprising this double issue relate to heat- and corrosion-resisting materials. The literature in this connection is as diverse in content as the multitude of applications found by nickel-base high-temperature alloys and the chromium nickel stainless steels, and there is space here to draw attention only to items covering a French Symposium on Aqueous and Gaseous Corrosion and to papers reporting studies of residual-oil-ash corrosion and the development of a C0-40 chromium-nickel casting alloy exhibiting improved resistance to this type of attack.

OF two data sheets recently issued by Elcontrol, Ltd., of Hitchin, Herts., the first (Data Sheet BC Issue 1) describes two current types of electronic counting and batching equipment, one type making use of Dekatron counting stages (BC1) and the other type (BC2) being a small self-contained batch counter for general industrial use at speeds up to 600 a minute. Data Sheet PR Issue 3, brings together a number of Elcontrol standard photoelectric units and describes a wider range of viewing heads and light sources than that contained in the original issue.

THE L. S. STARRET Co., Ltd., Jedburgh, Roxburghshire, which is a subsidiary of the well-known American manufacturers of precision tools and measuring instruments has published a brochure giving full technical details of its metal cutting band saw blades. Band saw blades in coil stock lengths and cut-to-size welded bands are some of the first products to come from the new 2 million dollar factory in the Scottish Borders. The booklet, which is attractively designed, contains tables showing blade widths, gauge or thickness, teeth per inch, and weight per 100 ft. for raker set, wavy set and skip tooth blades. A material-cutting recommendations table gives a useful guide to pitch selection, and a band saw trouble chart outlines several faults, caused by improper blade adjustment and the necessary remedies for correct operation of the cutting machine.

## Books Received

“Two Hundred Precious Metal Years: A History of The Sheffield Smelting Co., Ltd., 1760-1960.” By R. E. Wilson, 316 pp. inc. index; numerous illustrations. London, 1960. Ernest Benn, Ltd. 63s. net.

“The Practical Anodising of Aluminium.” By W. W. E. Hübner and A. Schiltknecht (translated by Winifred Lewis). 350 pp. inc. index, bibliography and appendix on testing methods; 187 illustrations. London 1960. Macdonald and Evans, Ltd. 55s. net.

“Overseas Newspapers and Periodicals.” Guide Book. H. R. Vaughan. 464 pp. London, 1930. Publishing and Distributing Co., Ltd. 30s.

“Metallurgical Reviews,” 1960, Vol. 5, No. 17. “The Theory and Practice of Vacuum Melting,” by O. Winkler; “Progress in Investment Casting,” by R. L. Wood. 135 pp. London, 1960. Institute of Metals. “Metallurgical Reviews”—a quarterly—is obtainable by annual subscription only: 50s. (\$7.50) post free; members of the Institute 32s. 6d. (\$5.00) post free).

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# LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

DECEMBER, 1960

Vol. LXII, No. 374

## Determination of Zirconium in Complex Nickel Alloys

By B. B. Bach, B.Sc., A.R.I.C., and J. T. Francis

(The Mond Nickel Co., Ltd., Development and Research Department Laboratory, Birmingham)

*A spectrophotometric method, using alizarin S, has been developed for the determination of zirconium in the range 0.05–0.2%. It is applicable to complex nickel alloys containing molybdenum, titanium, cerium, chromium, cobalt, aluminium and other elements.*

NICKEL alloys are widely used in the aircraft industry for applications involving service at high temperatures. Such alloys often contain major amounts of chromium (up to 20%), cobalt (up to 20%), molybdenum (up to 10%), titanium (up to 5%), and aluminium (up to 5%). In addition, iron, manganese, cerium, zirconium, copper and other elements may be present in amounts up to 0.5%. In alloys of these types zirconium can be determined spectrographically if a range of chemically analysed standards is available. The object of the work reported here was to develop a method for the determination of zirconium in such standards, covering the range from 0.01 to 0.2% Zr.

### Experimental Procedure

The usual gravimetric methods for determining zirconium, as phosphate,<sup>1</sup> oxide,<sup>1</sup> *p*-bromomandelate,<sup>2</sup> etc., were found to be insufficiently accurate for zirconium contents below 0.2%. Several colorimetric methods have been proposed using alizarin S,<sup>3</sup> morin,<sup>4</sup> quercetin,<sup>5</sup> phenylfluorone,<sup>6</sup> chloranilic acid,<sup>7</sup> catechol violet,<sup>8</sup> *p*-dimethylamino-benzene-azo-phenyl arsonic acid,<sup>9</sup> and (since this investigation was completed) xylenol orange.<sup>10</sup>

All the colorimetric methods mentioned above are suitable for small amounts of zirconium, but alizarin S was chosen as the most promising reagent because it was readily available, and because most of the interfering elements could be removed easily. Preliminary experiments, using the technique described by Mayer and Bradshaw,<sup>3</sup> with a pure zirconium chloride solution, showed that by plotting optical density (measured at 5250 Å on a Uvispek spectrophotometer) against zirconium concentration, a straight-line calibration graph could be obtained with quantities of zirconium ranging from 20 to 800 µg. in 100 ml. Above 800 µg., coagulation of the lake occurred, and below 20 µg. the graph was curved.

### Optical Absorption Curves

The absorption spectra of the zirconium alizarin S lake and of the reagents were measured on the Uvispek spectrophotometer. The curves are given in Fig. 1. The zirconium complex gives an absorption maximum around 5250 Å and at this wavelength the absorption due to the reagent is at a minimum.

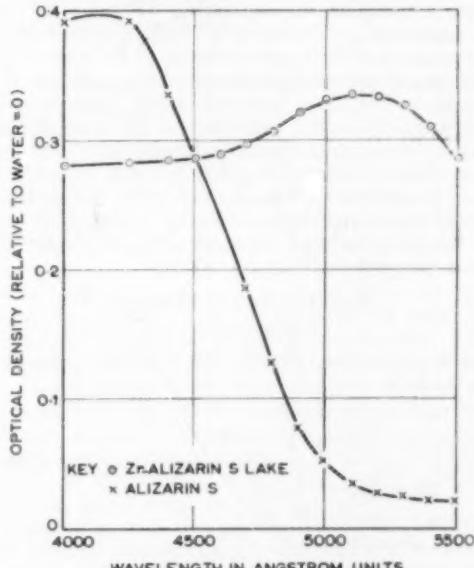


Fig. 1—Absorption curve of zirconium-alizarin S lake.

### Effect of Interfering Elements

Mayer and Bradshaw<sup>3</sup> list many of the relevant interferences, among them nitrate, fluoride, oxalate, phosphate, organic hydroxy acids (tartaric, etc.), large amounts of sulphate, oxidising agents, Fe<sup>III</sup> and Th<sup>IV</sup>. Among those not mentioned are Ti<sup>IV</sup>, Mo<sup>VI</sup> and Ce<sup>III</sup>, all of which can be present in nickel alloys. Tests showed that in amounts below 1 mg. Ce<sup>III</sup> did not interfere, but Ti<sup>IV</sup> interfered by giving cloudy solutions due to hydrolysis. Mo<sup>VI</sup>, when present in quantities around 50 mg., caused severe interference, as did large amounts of coloured ions such as Ni<sup>II</sup>, Cu<sup>II</sup>, Cr<sup>III</sup> and Co<sup>II</sup>. Contrary to Mayer and Bradshaw's finding, thioglycolic acid proved very satisfactory for the elimination of interference from small amounts of Fe<sup>III</sup>.

### Separation

From the interferences mentioned it was obvious that

TABLE I.—DETERMINATION OF ZIRCONIUM IN SYNTHETIC NICKEL-CHROMIUM-COBALT ALLOYS

| No. | Alloy | Zr Added (%) | Zr Found (%) | Average and Range (%) | Deviation from Added Zr        |
|-----|-------|--------------|--------------|-----------------------|--------------------------------|
| 1   | A     | 0.010        | 0.012        | 0.010                 | 0.011 $\pm$ 0.001 $\pm$ 0.001  |
| 2   | B     | 0.030        | 0.028        | 0.031                 | 0.029 $\pm$ 0.002 $\pm$ 0.001  |
| 3   | A     | 0.100        | 0.098        | 0.101                 | 0.10 $\pm$ 0.001 0 $\pm$ 0.002 |
| 4   | B     | 0.150        | 0.153        | 0.150                 | 0.152 $\pm$ 0.001 $\pm$ 0.002  |

Nominal composition of alloy type A : Co 20%, Cr 20%, Ti 2.5%, Al 1.5%, with less than 0.5% Si, Cu, Fe, etc., Ni balance.

Nominal composition of alloy type B : Co 20%, Cr 10%, Ti 1.5%, Al 5.0%, Mo 5%, with less than 0.5% Si, Cu, Fe, etc., Ni balance.

a preliminary separation of zirconium would be required. Electrolysis at a mercury cathode was known to be satisfactory for removing  $\text{Ni}^{II}$ ,  $\text{Co}^{II}$ ,  $\text{Fe}^{III}$ ,  $\text{Mo}^{VI}$ ,  $\text{Cr}^{III}$  and  $\text{Cu}^{II}$ , but  $\text{Ti}^{IV}$ ,  $\text{Al}^{III}$ ,  $\text{Ce}^{III}$  and some  $\text{Mn}^{II}$  would be left in solution. Of these elements,  $\text{Ti}^{IV}$  was the main interferant.

Various methods for separating  $\text{Ti}^{IV}$  and  $\text{Zr}^{IV}$  were tried, including precipitation of  $\text{Zr}(\text{OH})_4$  with ammonia in the presence of  $\text{H}_2\text{O}_2$ , precipitation of  $\text{Ti}^{IV}$  as a complex iodate, use of substituted arsonic acids, and use of ion exchange. The only method which gave complete recovery of  $\text{Zr}^{IV}$  was the extraction of  $\text{Ti}^{IV}$  as cupferrate<sup>11</sup> from a solution containing ethylenediamine-tetra-acetic acid (E.D.T.A.) buffered to pH 5, followed by extraction of  $\text{Zr}^{IV}$  as cupferrate from strong acid. Extraction of the cupferrates was more satisfactory than filtration of the precipitates because better recovery of  $\text{Zr}^{IV}$  and more efficient removal of  $\text{Ti}^{IV}$  was obtained.

### Recommended Method

#### REAGENTS

Unless otherwise stated, all reagents should be AnalalR grade.

*Nitric Acid* (sp. gr. 1.42).

*Hydrochloric Acid* (sp. gr. 1.16).

*Sulphuric Acid* (1:1).—Add 50 ml. sulphuric acid (sp. gr. 1.84) to 50 ml. water.

*Hydrogen Peroxide* (100 vol.).

*E.D.T.A. Disodium Salt*.

*Ammonium Hydroxide* (sp. gr. 0.880).

*Cupferron*.

*Chloroform*.

*Perchloric Acid* (sp. gr. 1.54).

*Thioglycollic Acid* (G.P.R. 1:1).—Add 50 ml. thioglycollic acid to 50 ml. water.

*Alizarin Red S*. (0.15% solution in distilled water).

*Zirconium Stock Solution* (1 ml.  $\equiv$  5 mg.).—Dissolve approx. 4 g. (see note) zirconium nitrate in 50 ml.  $\text{HNO}_3$  + 50 ml. distilled water and dilute to 250 ml. Standardise this solution by precipitating zirconium hydroxide with ammonia.

With a pipette, take 10 ml., dilute to about 15 ml., make ammoniacal, boil and filter. Ignite to  $\text{ZrO}_2$ , which contains 74.03% zirconium.

*Zirconium-Nitrate Working Solution*.—Dilute 1 ml. stock solution to 250 ml. with distilled water.

1 ml.  $\equiv$  20  $\mu\text{g}$ . Zr.

**N.B.**—This solution will not keep, as it hydrolyses. It should be freshly prepared immediately before use.

TABLE II.—DETERMINATION OF ZIRCONIUM IN SPECTROGRAPHIC STANDARDS

| No. | Alloy | Zr Found (%) | Average and Range (%) |
|-----|-------|--------------|-----------------------|
| 1   | A     | 0.014        | 0.015 $\pm$ 0.001     |
| 2   | A     | 0.017        | 0.017 $\pm$ 0.002     |
| 3   | A     | 0.022        | 0.023 $\pm$ 0.001     |
| 4   | A     | 0.048        | 0.044 $\pm$ 0.002     |
| 5   | A     | 0.094        | 0.094 $\pm$ 0.001     |
| 6   | B     | 0.017        | 0.018 $\pm$ 0.001     |
| 7   | B     | 0.027        | 0.029 $\pm$ 0.001     |
| 8   | B     | 0.030        | 0.030 $\pm$ 0.001     |
| 9   | B     | 0.030        | 0.031 $\pm$ 0.002     |
| 10  | B     | 0.037        | 0.036 $\pm$ 0.001     |
| 11  | B     | 0.049        | 0.045 $\pm$ 0.003     |
| 12  | B     | 0.090        | 0.088 $\pm$ 0.002     |
| 13  | B     | 0.128        | 0.128 $\pm$ 0.001     |

Nominal composition of alloy type A : Co 20%, Cr 20%, Ti 2.5%, Al 1.5%, with less than 0.5% Si, Cu, Fe, etc., Ni balance.

Nominal composition of alloy type B : Co 20%, Cr 10%, Ti 1.5%, Al 5.0%, Mo 5%, with less than 0.5% Si, Cu, Fe, etc., Ni balance.

**Note**—Zirconium nitrate has a variable composition and does not conform to the formula  $\text{Zr}(\text{NO}_3)_4$ . When this work was carried out 4 g. was found to be correct for the sample available, but that amount may not be correct for samples from other suppliers.

#### PROCEDURE

Dissolve 1 g. (see Note 1) of drillings in 50 ml. 1:1 hydrochloric acid/nitric acid. Add 4 ml. 1:1 sulphuric acid and evaporate cautiously to fumes of sulphur trioxide. Do not heat too strongly (see Note 2). Add 40 ml. water and heat gently to dissolve salts. Filter hot through a Whatman 541 paper and wash with hot water. Reserve the residue (see Note 3). Transfer to a mercury-cathode electrolysis cell and electrolyse at 4–5 A. (see Note 4) until the solution is colourless. Wash-in the sides of the cell and continue the electrolysis for a further 30 minutes. Run off the solution from the mercury and wash the cell with water.

Add 1 ml. hydrogen peroxide (100 vol.) (see Note 5), and evaporate the solution just to fumes of sulphur trioxide. Allow to cool and add 25 ml. water. Transfer to a 150 ml. separating funnel and dilute to approximately 50 ml. Add 2 g. E.D.T.A. and shake until dissolved. Add ammonium hydroxide to make just alkaline to methyl orange, and cool thoroughly. Add approximately 0.3 g. cupferron and 10 ml. chloroform; shake well. Allow the yellow chloroform layer to separate and discard it. Repeat the extraction twice, using approximately 0.3 g. cupferron and 10 ml. chloroform each time. Finally, extract twice with 10 ml. portions of chloroform. The last chloroform extract should be colourless.

Add 10 ml. hydrochloric acid and cool thoroughly. Add approximately 0.3 g. cupferron, extract with 3  $\times$  10 ml. portions of chloroform, and combine the extracts in a 100 ml. beaker. Evaporate the chloroform carefully until a brown oily liquid is left. Very cautiously add approximately 20 ml. nitric acid, taking precautions to prevent loss of solution by effervescence. Add 3 ml. perchloric acid and evaporate cautiously to fumes of perchloric acid. Remove the beaker cover and evaporate just to dryness.

Add 3 ml. hydrochloric acid, boil and then add 10 ml. water and 3 drops 1:1 thioglycollic acid. Heat to about 80°C. and add 10 ml. 0.15% alizarin Red S solution. Place in a boiling-water bath for 3 minutes, cool to room

temperature and dilute to 100 ml. Read the optical density at 5250 Å on a Uvispek spectrophotometer (see Note 6), using a 4 cm. glass cell, against a blank solution consisting of 3 ml. hydrochloric acid, 10 ml. water, 3 drops thioglycolic acid and 10 ml. 0.15% alizarin Red S, which has been heated in the water bath as above.

Read off the zirconium content from the calibration graph.

#### NOTES

**Note 1.**—The optimum quantity of zirconium in the final solution lies between 100 µg. and 500 µg. although satisfactory results can be obtained from 20 µg. to about 800 µg. For zirconium contents higher than 0.05% proportionately smaller samples should be taken.

**Note 2.**—If the salts are heated too strongly there is a danger that insoluble chromium salts will be formed.

**Note 3.**—If zirconia ( $ZrO_3$ ) is suspected in the alloy this residue should be fused with potassium bisulphate and the extract added to the original solution before electrolysis.

**Note 4.**—Using an uncooled cell, 4–5 Å. is the maximum current allowable, but this can be increased if water-cooled cells or electrodes are available.

**Note 5.**—The hydrogen peroxide is added to prevent hydrolysis of titanium salts. When the titanium content is low this step can be omitted and the solution can be simply evaporated to about 30 ml.

**Note 6.**—Other spectrophotometers could be used, or the Spekker Absorptiometer should be satisfactory, using Ilford yellow-green filter No. 605 and H.503 heat filter.

#### CALIBRATION GRAPH

Add known amounts of zirconium nitrate solution to suitable weights of either an alloy containing no zirconium or of a synthetic alloy solution prepared from salts. Analyse these samples by the procedure given, and plot a curve relating optical density to micrograms of zirconium. The zirconium additions should range from about 20 to 800 micrograms and the weights of alloy used should be appropriate to the zirconium contents of the samples to be analysed. The composition of the synthetic alloy should approximate to that of the samples.

#### Discussion

Throughout this investigation no account was taken of the presence of hafnium, which is usually present in commercial zirconium in amounts of 1–3%. Mayer and Bradshaw report that under the same conditions hafnium forms a similar lake, but the absorption is only one quarter of that given by zirconium. The effect of hafnium will therefore be negligible.

When the zirconium : titanium ratio was too low, difficulty was experienced in obtaining a clean separation: satisfactory results were, however, obtained down to 0.01% Zr in the presence of 3.5% Ti. The method has been found to be satisfactory from about 0.01% to 0.5% Zr: above about 0.2% Zr the gravimetric method, using *p*-bromomandelic acid, is more accurate.

A selection of results obtained by this method, when used on nickel-cobalt-chromium alloys containing molybdenum, aluminium and titanium, is given in Table I.

#### Acknowledgment

The authors are indebted to The Mond Nickel Co., Ltd., for permission to publish this paper.

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## Correspondence

### RAPID METHODS OF ANALYSIS

The Editor, METALLURGIA.

Sir,

Analytical control of metallurgical processes such as the production of alloys and the refining of metals is most conveniently and rapidly achieved using one or other form of direct reading spectrophotograph. Such instruments are by no means available to every control chemist, however, and there is still a need for wet chemical methods which will produce results of adequate accuracy in as short a time as possible—preferably in not more than fifteen minutes from the time of taking the sample.

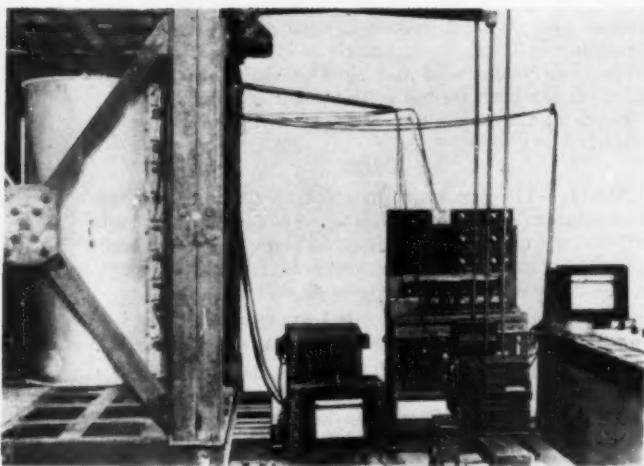
It is perhaps not widely realised that the biggest obstacle to achieving analysis times of this order is the time taken to effect solution of the sample. This is illustrated by a composite photometric determination of iron, copper and nickel in an aluminium alloy in which the stages of the operation were timed. When the sample (200 mg.) consisted of millings or drillings of the size normally employed it was found that of the total analytical time of 26 minutes 20 seconds, dissolution of the sample (in sodium hydroxide solution) took 17 minutes 40 seconds, i.e. over two thirds. When the sample was in the form of sawings the same amount was dissolved in the same caustic solution in times ranging from 3 to 7 minutes—a saving of nearly 11 to 15 minutes.

Sawings have the further advantage that they can be obtained rapidly and simply, without the use of power operated machinery—only a vice and a hack-saw are required—and the sawings are representative since cuts can be made through the whole cross-section of the sample casting. In practice a flat chill casting, preferably  $\frac{1}{2}$  in. to  $\frac{3}{4}$  in. in thickness, is cut to the centre line a number of times from both edges using an ordinary hacksaw with a 12-in. blade having 18 teeth to the inch. Sufficient sample (freed of iron particles by a magnet) can be obtained in this way within three minutes.

That analytical time can be saved by using a fine sample is obvious, but the above quantitative illustration of the order of saving which can be achieved with aluminium alloys, and which becomes of greater significance for quick methods, may be of interest to your readers. It is one of the findings which resulted from a critical examination of rapid methods available for the

(continued on page 284)

# Accurate Temperature Control for Creep/Rupture Testing Installation



The first of the six furnaces with the switch gear panel located between the Honeywell strip chart recorder-controller on the right and the recorder in the centre foreground.

ELECTRIC furnace zone control to within  $0.5^{\circ}\text{C}$ , or closer, has been achieved at a new creep/rupture testing installation at the Research Department of Colvilles, Ltd., at Mossend, nr. Glasgow. This is believed to represent finer control of temperature than has been obtained hitherto in this type of work. When complete the installation will comprise six new electric resistance furnaces, which will be used to collect creep/rupture data on various steels which Colvilles produce. Each furnace will operate at a different temperature, the six temperatures being  $400^{\circ}\text{C}$ ,  $450^{\circ}\text{C}$ ,  $475^{\circ}\text{C}$ ,  $500^{\circ}\text{C}$ ,  $525^{\circ}\text{C}$ , and  $550^{\circ}\text{C}$ , all  $\pm 10^{\circ}\text{C}$ . The temperature control is well within the tolerances laid down by the appropriate British Standards governing this type of test.

## Continuous Record

Each furnace is itself divided into six zones containing thermocouples; thermocouples are also placed adjacent to the specimens. Using Colvilles' own switchgear panel, measurements of these points are taken in sequence, thermocouples being connected for a few seconds each. A continuous record is given on a Honeywell Controls Electronik strip chart recorder-controller; the ink trace is continuous since there is an 'overlap' in the switchgear which provides the average of the temperature of two zones when switching over from one zone to another.

The Electronik instrument acts as a controller in conjunction with the switchgear panel, and experience with the first furnace showed a maximum variation of  $\frac{1}{2}^{\circ}\text{C}$  per zone and  $1\frac{1}{2}^{\circ}\text{C}$  over the whole furnace under the worst combination of errors. Such precision may in large measure be attributed to the continuous method of control. The recorder-controller operates on the self-balancing potentiometer principle. A voltage representing the furnace temperature is applied to a slidewire and is compared to a standard voltage; any departure from the required temperature is conveyed through an amplifier system to a motor which moves the slidewire contact and places the circuit in balance again. At the same time the ink trace changes position laterally and

the switchgear panel is used to change the supply to the furnace zone.

## Additional Safety

An additional safeguard covering the whole circuit is provided by means of a Honeywell Protect-O-Vane excess temperature safety cut-off. The creep/rupture tests are carried out over extended periods, and, although low temperature could be tolerated, a single instance of the temperature rising above the preset value would ruin the test and waste much time. This overall protection in the case of each furnace has therefore been thought prudent. The Protect-O-Vane is arranged to operate from two thermocouples in series. Finally, a further item of instrumentation is an extra Electronik which is a strip chart recorder only, for occasional study of selected thermocouple readings.

## Mass Spectrometers for Russia

An order value £50,000 has been received by the Instrumentation Division of Associated Electrical Industries, Ltd., from a London confirming house for two MS7 spark-source mass spectrometers for Russia. The spectrometers will be manufactured at the A.E.I. Trafford Park Works and will be delivered to V/O Mashpriborintorg U.S.S.R.

## Correspondence

(continued from 283)

analysis of aluminium alloys made recently by the Chemists' Sub-Committee of the Association of Light Alloy Refiners and Smelters.

Yours faithfully,  
F. H. SMITH,

Technical Officer

The Association of Light Alloy Refiners and Smelters, Ltd.,  
3, Albemarle Street, London, W.1.

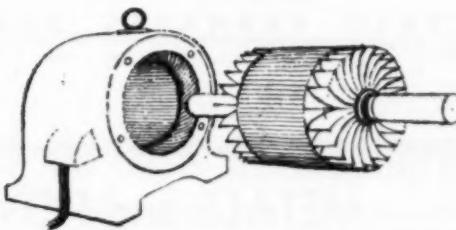
November 1960

## Electric Motors & Controls 1

Most manufacturers today employ electric motive power through individual drives, which, among their many advantages, permit the right type of motor to be used for each of various types of machine. The range of motors available—each with its own characteristics—is very large, and the factory executive could well be guided in his choice by the expert views of the motor manufacturer, the installing engineer or his Electricity Board's engineer. The characteristics of the main types of motor are briefly summarised below.

### Squirrel-cage Motors

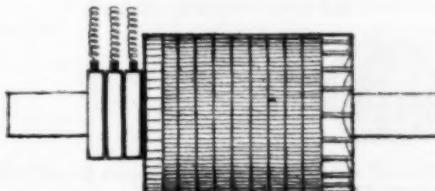
These are the most straightforward and simple in design, and are therefore relatively cheap and robust in character. They should be considered for general duties and, in conjunction with variable-speed gears or couplings, for applications requiring variable speeds, e.g. for crane drives. Small sizes can be switched direct-on-line.



The squirrel-cage motor is very suitable for individual drive of each motion of single-purpose machine tools where the motor horsepower can be precisely specified. It is suitable for driving pumps, fans, lifting hoists and woodworking machines. Textile machinery represents another field of use.

### Slip-ring Motors

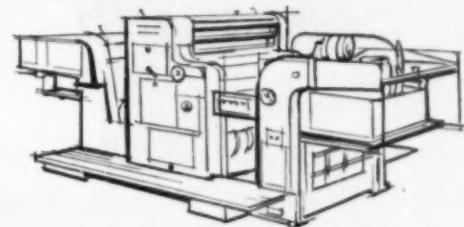
The chief advantage of the slip-ring motor is a very low starting current for a given torque, e.g. full-load torque at starting with a current about 10% above full-load current. This makes it suitable for applications requiring a prolonged starting period with a



load of high inertia. It also permits of speed variation below synchronous speed, though with some loss of efficiency. Typical applications include fans, pumps, heavy lathes, grinders and boring mills, as well as calendering machines, cable-laying-up machines and mine hoists.

### Three-phase A.C. Commutator Motors

The main characteristic of this type is variable-speed with uniform and gradual acceleration and good efficiency over the speed range. Paper manufacture provides excellent examples of its use, e.g. in paper-making, reeling, cutting, calendering, coating and drying.



A.C. commutator motors are recommended for mechanised bakeries and for cranes and hoists where very slow speeds are frequently needed.

### Synchronous Motors

These are constant-speed motors. One particular advantage is that they can be operated at unity or even at leading power factor to correct a system suffering from lagging power factor, and perhaps so qualify for a reduction in the electricity bill. Pump and compressor drives are typical uses to which they can be put. They are also used in motor-generator sets.

### Single-phase A.C. Motors

In general, single-phase motors are used in light industries for drives not requiring more than about five horsepower or where a three-phase supply is not available. While their use is mainly limited to work of a light nature, they do fill a need in such duties as sewing-machine drives, portable hand tools, window opening and closing gear, etc.

### Direct Current Motors

For a completely unfettered performance where wide ranges of speed variation, high rates of acceleration and powerful dynamic braking are all-important, the D.C. motor is unrivalled. This means in effect the installation of an A.C.-D.C. motor-generator set or rectifier to give the necessary supply, but the increased cost may well be compensated by the improvement in productivity. When variable voltage is applied to the armature, a wider speed range is obtainable than with any other type of motor. Typical applications of the D.C. motor are: cranes, haulage and tippler equipment, certain machine tools requiring a large speed range and smooth acceleration, high-speed printing and steelworks drives.

For further information, get in touch with your Electricity Board or write direct to the Electrical Development Association, 2 Savoy Hill, London, W.C.2. TEMple Bar 9434.

They can offer you excellent reference books on electricity and productivity (8/6, or 9/- post free)—"Electric Motors and Controls" is an example.

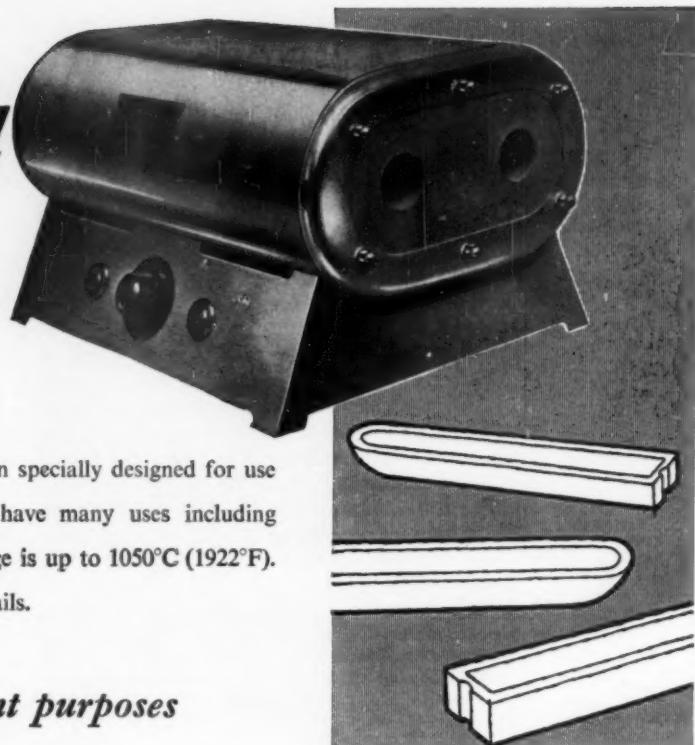
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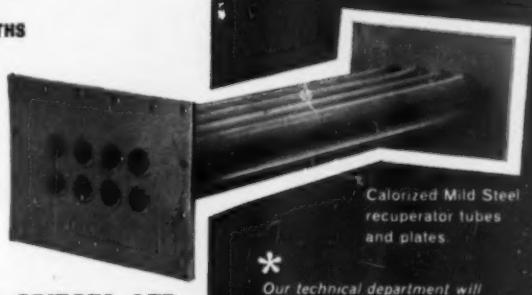
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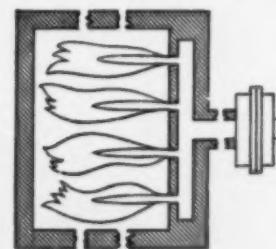
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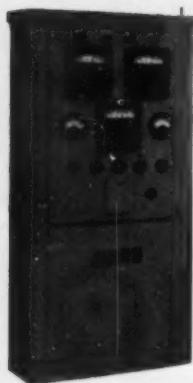
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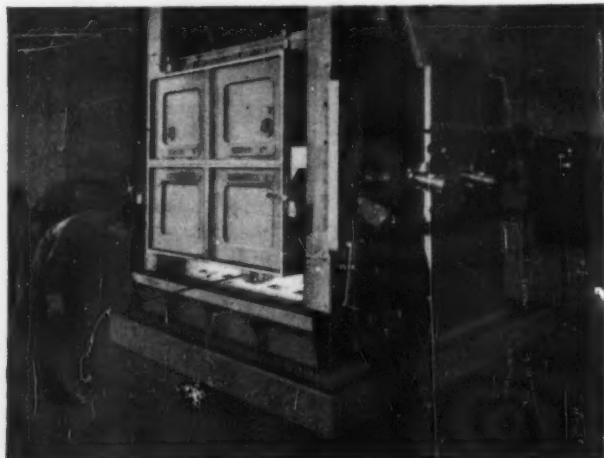
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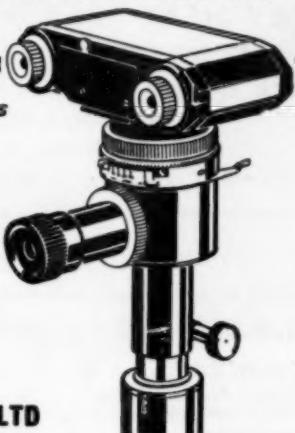
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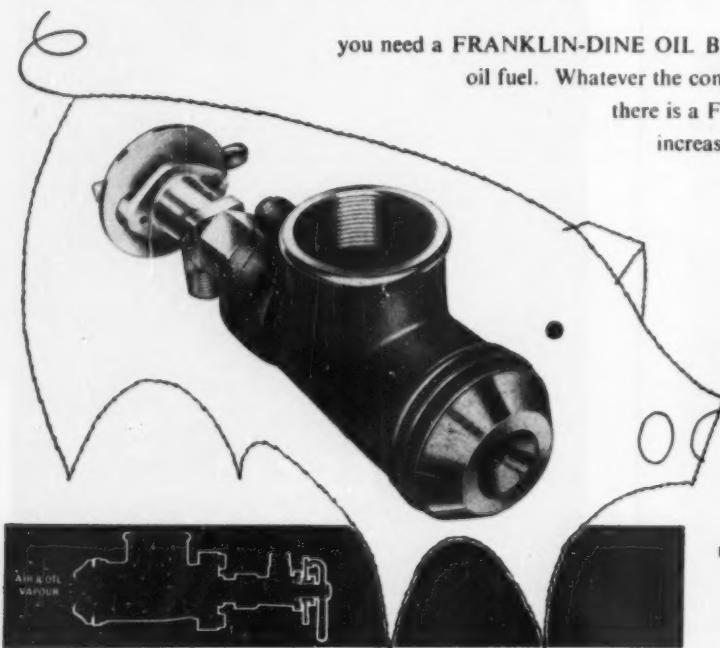
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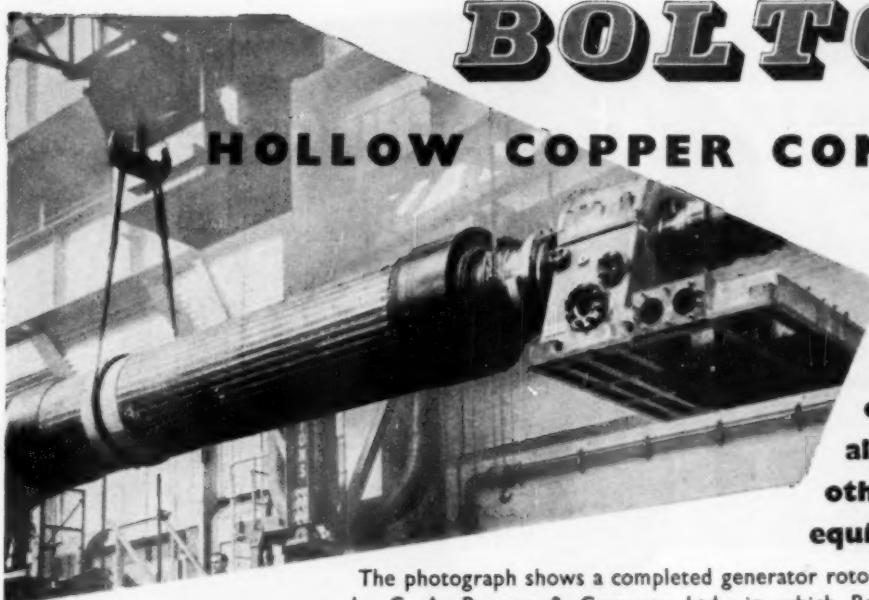
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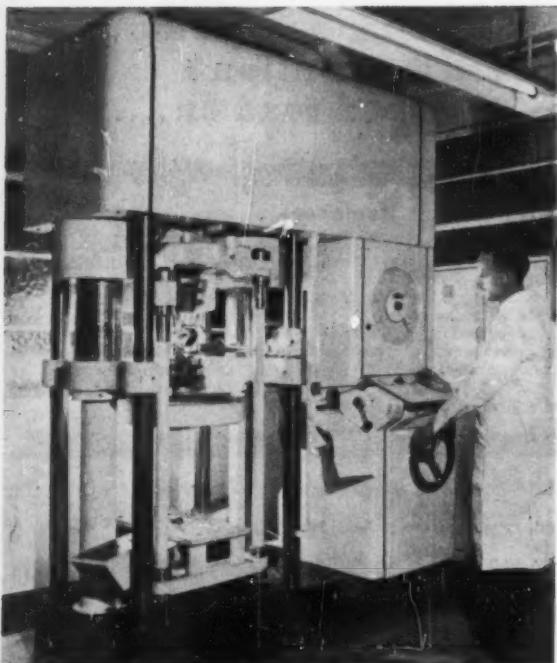


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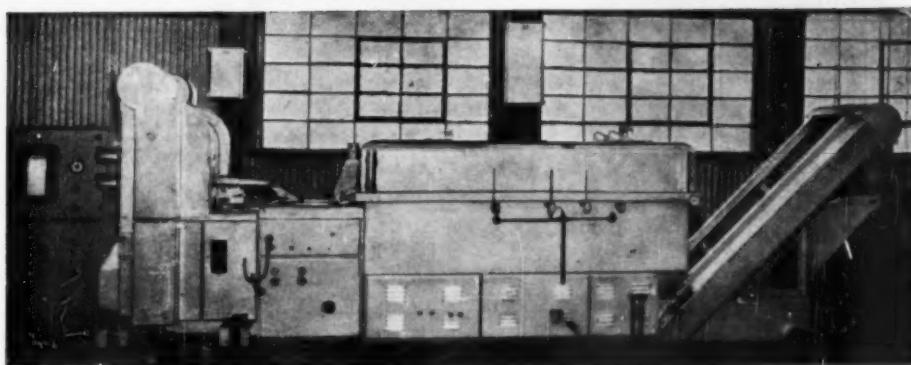
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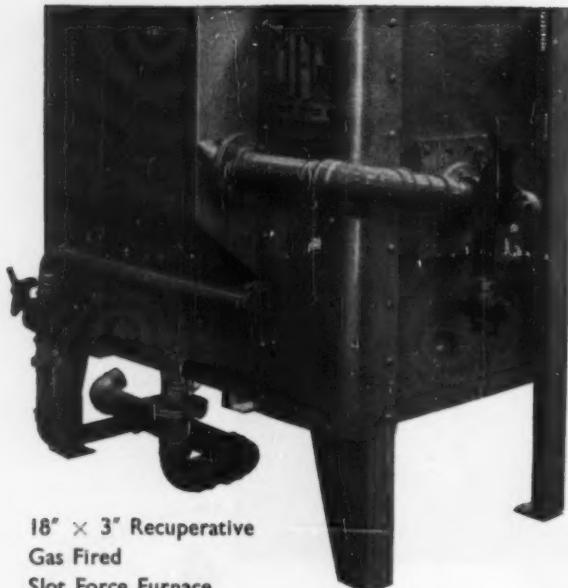
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Whilst the use of standard measuring instruments and gauges remains common practice in production engineering, important advances have been made in recent years and the time seems opportune for putting the now numerous methods into a reference framework, as it were, so that their scope and relationship may be readily appreciated. Accordingly in this edition of the **MECHANICAL WORLD YEAR BOOK** we present a series of tables and notes summarising under specific headings the available methods for direct and comparative measurement.

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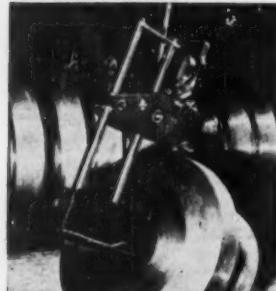
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